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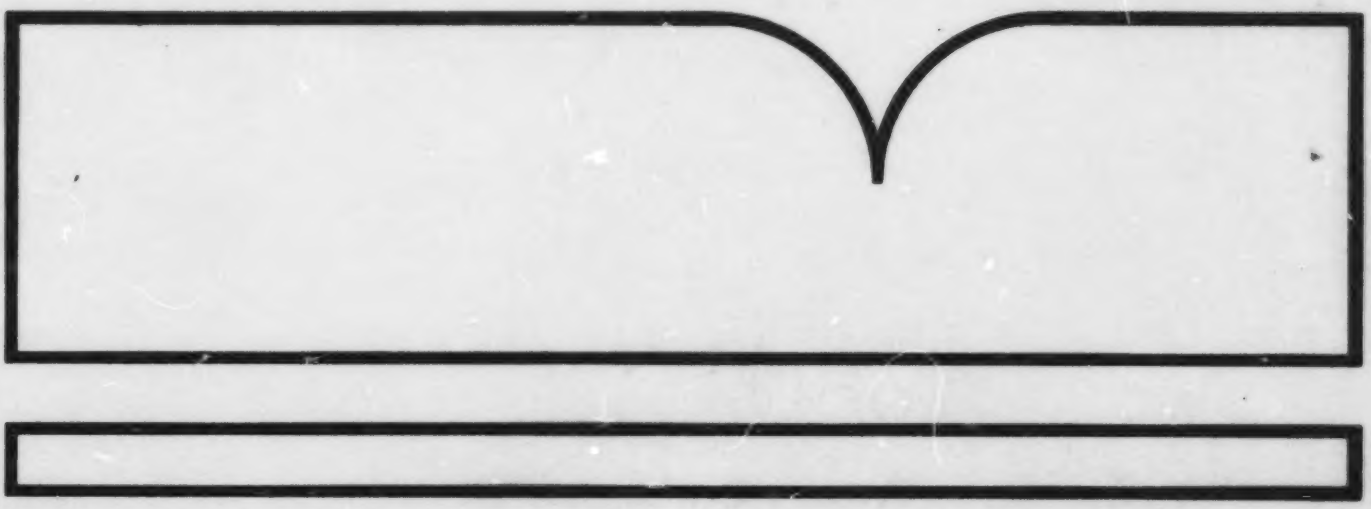
Securing Containerized Hazardous Wastes by Encapsulation With Spray-On/Brush-On Resins

H. R. Lubowitz, et al

Environmental Protection Polymers, Incorporated
Hawthorne, California

July 1981



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SECURING CONTAINERIZED HAZARDOUS WASTES BY ENCAPSULATION WITH SPRAY-ON/BRUSH-ON RESINS

by

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Contract Nos. 68-03-2483 and 68-03-2993

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16. ABSTRACT

Methods were investigated for securing containerized hazardous wastes in the field with spray-on or brush-on resins at ambient temperatures. Laboratory-scale, cylindrical specimens of containerized sodium chloride (which simulated soluble salts containing heavy metals) were fabricated 63.5 mm in diameter by 88.9 mm high (2.5 by 3.5 in.). The salt was confined in fiberglass-reinforced, thermosetting resin castings and then sealed with a water-based polyurethane to provide a tough exterior jacket that would protect the contents from leaching and mechanical stress.

The specimens exhibited functional stiffness and kept their contents stable when subjected to leaching stresses. The exterior jackets did not fail when specimens were compressed to about 80% of their original heights. Under heavy compression, however, the jackets ruptured where they were thinnest. Several coatings were needed to produce sufficiently thick, tough jackets.

This report is a companion to two other documents on the use of plastics to encapsulate corroding hazardous waste containers: Securing Containerized Hazardous Wastes with Polyethylene and Fiberglass Encapsulates (EPA-600/) and Securing Containerized Hazardous Wastes with Welded Polyethylene Encapsulates (EPA-600/).

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

This report discusses one of several methods investigated to make leaking containers of hazardous waste safe for disposal or long-term retention. Spray-on or brush-on techniques were used to encapsulate the containers with a polymeric resin under ambient conditions.

Francis T. Mayo, Director
Municipal Environmental Research
Laboratory

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ABSTRACT

Methods were investigated for securing containerized hazardous wastes in the field with spray-on or brush-on resins at ambient temperatures. Laboratory-scale, cylindrical specimens of containerized sodium chloride (which simulated soluble salts containing heavy metals) were fabricated 63.5 mm in diameter by 88.9 mm high (2.5 by 3.5 in.). The salt was confined in fiberglass-reinforced, thermosetting casings and then sealed with water-based polyurethane to provide a tough exterior jacket that would protect the contents from leaching and mechanical stress.

The specimens exhibited functional stiffness and kept their contents stable when subjected to leaching stresses. The exterior jackets did not fail when specimens were compressed to about 80% of their original heights. Under heavy compression, however, the jackets ruptured where they were thinnest.

The major shortcoming encountered in the use of the polyurethane was the need for several coatings to produce sufficiently thick, tough jackets. One or two applications were found to be sufficient to seal the specimens effectively, but they were not thick enough to yield jackets that would remain functional under heavy compression.

This report is a companion to two other documents investigating the use of plastics for the encapsulation of corroding containers of hazardous wastes: Securing Containerized Hazardous Wastes with Polyethylene and Fiberglass Encapsulates (EPA-600/) and Securing Containerized Hazardous Wastes with Welded Polyethylene Encapsulates (EPA-600/).

This report was submitted in fulfillment of Contract No. 68-03-2483 by TRW Defense and Space Systems Group and Contract No. 68-03-2993 by Environmental Protection Polymers, Inc., under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period 1978 to 1980, and work was completed as of December 1980.

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ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

ASTM	-- American Society for Testing and Materials
^{o}C	-- degrees Celsius
cps	-- centipoise
CS	-- "Calibrase" wheel designation
cyc	-- cycles
DI H_2O	-- deionized water
DOT	-- Department of Transportation
^{o}F	-- degrees Fahrenheit
g	-- grams
g/cm^3	-- grams per cubic centimeter
hr	-- hour
in.	-- inch
IPA	-- isopropyl alcohol
kN	-- kilonewton
KOH	-- potassium hydroxide
lb	-- pound
lb/gal	-- pounds per gallon
mg	-- milligrams
mil spec	-- military specification
ml	-- milliliter
ml/min	-- milliliter per minute
mm	-- millimeter
MPa	-- megapascal
N	-- normality of solution
NaCl	-- sodium chloride
Pa·s	-- pascal·second
pbw	-- parts by weight
PE	-- polyethylene
pH	-- $-\log [H_3^+O]$
ppm	-- parts per million
psi	-- pounds per square inch
RH	-- relative humidity
rpm	-- revolutions per minute
TiO_2	-- titanium dioxide

SYMBOLS

C	-- carbon	O	-- oxygen
H	-- hydrogen	~~~~	-- polymer backbone
N	-- nitrogen	X	-- number of repeated groups in polymer

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Our thanks are also extended to Mrs. V. R. Butler for providing clerical services.

SECTION 1

INTRODUCTION

This study investigates on a laboratory scale methods and materials for securing containerized hazardous wastes in the field with spray-on or brush-on resins at ambient temperatures. Cylindrical specimens of containerized sodium chloride (which simulated soluble salts containing heavy metals) were fabricated 63.5 mm in diameter by 88.9 mm high (2.5 by 3.5 in). The salt was confined in fiberglass-reinforced thermosetting resin casings and then sealed with water-based polyurethane to provide a tough exterior jacket that would protect the contents from leaching and mechanical stress.

Evaluations of encapsulate specimen performance concerned mainly their ability to prevent delocalization of their sodium chloride consignments in leaching by water, and to withstand heavy compression without rupture of their jackets. Retention of the highly soluble sodium chloride was assumed to indicate similar encapsulate performance for soluble heavy-metal-containing salts. It was also assumed that specimens compressed heavily without jacket rupture would also be able to withstand severe impact. (Although the jacketing resin per se was expected to withstand impact because of its rubber-like qualities, it was subject to additional stresses stemming from distortion of the specimens under compression and fragmentation of the stiff fiberglass materials.)

The purpose of this work was to set forth means for encapsulating containerized hazardous wastes (with particular emphasis on 208-liter [55-gal] drums) by employing the simple techniques of spraying or brushing materials at atmospheric temperatures onto the surfaces of the containers. Spraying and brushing are desirable techniques because low-cost, commonly used equipment can be used. Processing at atmospheric temperatures had obvious advantages. The coating materials were required to consolidate (i.e., spread and cure) on the container surfaces and to give rise to monolithic, tough, chemically stable encapsulates. The encapsulates were expected to hold their hazardous consignments securely when subjected to mechanical and chemical stresses associated with sequestering them in disposal sites.

The following factors may affect encapsulates:

- Impact and compression during transit.
- Tear and overburden resulting from onsite processing and depositing in ultimate disposal sites.

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- Disposal site leachates.

- Corrosive action of hazardous consignments.

Structuring encapsulates to withstand impact and compression stresses was particularly difficult. According to DOT guidelines, containers geared for transporting hazardous wastes must withstand the test of dropping on edge without rupture. Since a 208-liter (55-gal) drum and its contents weigh about 226.8 kg (500 lb), such a test would induce severe tensile and flexural stresses on the encapsulates, thus requiring the use of tough, flexible materials for container fabrication. Furthermore, encapsulates must be composed of stiff materials to withstand compression stresses resulting from the stacking of heavy objects. (Other stresses were less difficult to compensate for.) Pressures exerted by landfill overburdens were not particularly severe when encapsulates were deposited deeply enough for the soil to act as a fluid. Encapsulate resistance to chemical degradation did not need to be characterized in detail because it could be estimated by data from resin vendors and performance data provided in the chemical literature. Permeability resistance data for non-mass-produced resins were not readily available, and thus some determinations were required.

No spray-on or brush-on material was currently available for encapsulate fabrication that could provide the required impact and compression resistance as well as chemical stability. Metals and ceramics were ruled out. Resins, on the other hand, gave rise to difficult to resolve problems: impact resistance advantages were usually at the expense of stiffness, and toughness was related to processing parameters (unfortunately, state-of-the-art resins employed in spray-on and brush-on applications were as a class weak materials). Production of stronger materials required the use of newly developed resins.

Resins formulated for spray-on and brush-on use were further evaluated with respect to the purpose of this work and to environmental regulations. In general, such resins contained either solvent or no solvent. Non-solvent-containing resins were obviously desirable, but these were two-component reactive systems that are difficult to manage under field conditions. Solvent-containing resins were more desirable one-component systems of two major classes: Either they contained organic solvents or water. But those with organic solvents were disallowed by environmental regulations; they presented potential hazards associated with their use. Consequently, the resins employed in this work were water-based.

Although water-based polyurethanes exhibited appropriate toughness and flexibility to withstand impact stresses, they did not possess the stiffness required for encapsulates. Toughness and stiffness were realized by fabrication of composite structures, which provide properties not found in a single material. In this work, fiberglass-reinforced epoxides were used to yield stiff materials to provide encapsulates with required functional stiffness. Furthermore, they could be sprayed onto substrates using commercial techniques.

The fiberglass casings were constructed first during specimen fabrication. Because stiffness only was required, there was no need to realize the watertight structures that are sought in plastics fabricating. Sealing of the casings would be effected by applying water-based polyurethane resin onto the stiff materials.

In addition to sealing the encapsulates, the polyurethane resin was expected to provide them with impact resistance. (Structuring stiff materials per se to withstand severe impact stresses without fracture would be prohibitive in terms of material and processing costs. Consequently, the stiff materials would provide functional stiffness and impact resistance only under normal handling conditions; but under severe stresses, they were expected to fracture.) The hazardous contents were expected to remain secure as a result of the presence of the tough sealing resin. Even with fracture, the encapsulates were expected to exhibit sufficient residual stiffness to allow manipulation to and deposition within a direct disposal site.

The determining criteria for estimating suitable performance of encapsulates under field conditions was the satisfactory performance of the test specimens under heavy compression. The sealing resin had to exhibit the property of withstanding the stresses without rupture stemming from the fracture of the stiff materials. Such performance would indicate that the sealing resin would secure the hazardous consignments in the event that the encapsulates were subjected to severe impact stresses.

SECTION 2

CONCLUSIONS

The fabrication of viable encapsulates of containers holding hazardous wastes under atmospheric conditions employing the simple means of brushing or spraying materials onto the surfaces of containers is feasible by use of the resins tested here. The watertight encapsulate materials studied here are considered to be unique because they are stiff but retain their watertight properties when distorted by compression. Generally, only metals yield such properties, but they are prone to corrosion.

The use of fiberglass-reinforced resins (epoxides) and water-based polyurethane resins gives rise to room-temperature-cured encapsulates that keep their water soluble salt consignments stable under aqueous leaching conditions. The application of the polyurethanes to the surfaces of fiberglass/epoxide substrates effectively seals porosity existing in the substrates. Consequently, watertight double-walled composites are obtained, with the outer wall acting as a jacket for the encapsulates.

The polyurethane resins that exhibit best performance are those containing chemical cross-linking agents. The curing of the polyurethane resins on surfaces of preformed glass/epoxides yield double-wall composites showing excellent resistance to hydrolysis. Thereby the sealing effect of the polyurethane jackets is assured. Since the polyurethane resin per se was not expected to transpire salt, this work showed that the high salt retention performance of encapsulates (i.e., double-walled composites) is due to wetting and sealing by water-based polyurethane resin in fiberglass/epoxide substrates and forming of hydrolytically stable bonds between the applied resins and the substrates.

The fiberglass/epoxide substrates give expected stiffness to encapsulates. With compression of the encapsulates, the substrates yield, as expected, by fracture. The encapsulate's load-bearing properties under heavy compression were due to the residual strengths of the fractured substrates and the existence of the polyurethane jackets.

The jackets do not show satisfactory performance under heavy encapsulate compression (i.e., greater than 20%). Rupture of the jackets was the observed mode of jacket failure. To be sure that full-scale encapsulates would satisfy DOT guidelines concerning transportation of containerized hazardous wastes, it was assumed that jackets should remain functional under very heavy (at least 80%) encapsulate compression. Thus the present development of this work does not assure realization of full-scale encapsulates that would comply with transportation regulations.

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This study does, however, provide means for securing the contents of hazardous-waste-containing, corroding containers at the site of their disposition. Containers threatening to lose their contents can be reinforced on the spot by simple means. The entire container or the greatly corroded areas can be effectively treated by applying fiberglass/epoxide to the surface (glass mat applied to container, then wetted by epoxide spray would be desirable) followed by application of water-based polyurethane resin.

The conclusions reached by this study are as follows:

- Fabrication is readily realized under atmospheric conditions employing simple means (e.g., spraying and/or brushing on resins and employing fiberglass, possibly either as mat or as sprayable, chopped fibers).
- High performance retention of heavy metal salts can be expected based on results of water leaching of encapsulates holding sodium chloride as a waste simulant.
- Thicker jackets must be realized for assuring compliance with DOT guidelines.

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SECTION 3

RECOMMENDATIONS

Advancement of this work requires bench-scale development to achieve thick coatings with one or at most two applications. This additional work would be necessary to fabricate encapsulates of 55-gal drums of hazardous wastes that can meet DOT guidelines for containers used to transport hazardous materials.

The present status of the work permits ready refinement for application to the sealing of 55-gal drums of hazardous materials that have undergone appreciable corrosion. This remedial action would secure the contaminants until provision is made for their final disposition.

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SECTION 4

MATERIALS

The identification of resins offering physical and chemical properties desirable for high-performance jackets was limited by the scarcity of those that cure at atmospheric temperatures and give rise to tough, flexible coatings. Atmospheric-temperature-curing coatings commonly employed result in brittle, rather than flexible products. These are serviceable if the applied-to surfaces are not subjected to significant amounts of flexure. (Plastics technology constantly strives to mitigate this deficiency.) In the absence of substrate surfaces, they exhibit minimal useful mechanical properties. Since these properties are not consistent with those needed for high performance jackets for encapsulates, the bulk of commercially available atmospheric-temperature-curing resins were ruled out.

State-of-the-art thermoplastic (but not atmospheric-temperature-curing) polyurethanes were considered to be a model polymer class during the selection process because of their overall balance of properties: high tensile strengths, excellent abrasion resistance and resistance to solvents, excellent adhesion to most types of surfaces, good elasticity, low permeability, and good resistance to weathering.[1] (These coatings have many commercial applications, such as linings for storage tanks on seagoing tankers hauling crude oil in one direction and salt water during return trips, and conveyor systems that haul corrosive and abrasive materials.[2])

CANDIDATE COATING MATERIALS

Review of the literature, with the aforementioned in mind, led to the disclosure of a promising novel generation of atmospheric-temperature-curing polyurethanes without the deficiencies described earlier. Four coating formulations were subsequently selected for screening as potential candidates for jacket studies (Table 1).

SCREENING STUDIES

The screening procedure included tensile, hardness, immersion tests, and general observations, with the following goals in mind:

- To provide evidence indicating that the jacket formation process does not significantly alter the anticipated properties of the polyurethane coating.

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TABLE 1. FOUR COATING FORMULATIONS* SELECTED FOR SCREENING STUDIES

Composition	Formulation (parts by weight)
Neorez R-960	100
Neorez R-960 Cross-linker CX-100	100 3
Neorez R-962	100
Neorez R-962 Cross-linker CX-100	100 3

*Vendor - Polyvinyl Chemical Industries, Wilmington, Mass.

- To investigate the general processability of each formulation.
- To advance the coating formulation best suited for detailed encapsulation studies.

Tensile Tests

Specimens of each coating, reinforced by fiberglass casings,* were prepared and cured for 4 days at 23° to 24°C. Specimens were tested for tensile properties on an Instron Universal Testing Instrument, model 1130, in accordance with ASTM-D-412 (Appendix A).

Stress-strain graphs showing response modes of tensile specimens are given in Figures 1 through 4. The fiberglass casings contribute rigidity in initial extension as shown by the discontinuity (spiked portion at ~3% strain) in each graph. Upon failure of the casings, the stress is transferred to the coatings, and remaining properties reflect the unsupported jackets in tension. Table 2 contains the quantitative data obtained from this study.

Comparison of Figures 1 through 4 and review of Table 2 indicated R-960 and R-960/CX-100 to be the two toughest coatings as judged by their stress-strain behavior. The primary differences between the two involved better ductility for R-960, but enhanced chemical resistance was reported for R-960/CX-100.

*Fiberglass casings were prepared from glass cloth impregnated with ~40% (by weight) epoxy resin (Epon 828, diethylenetriamine), which cured at room temperature.[3]

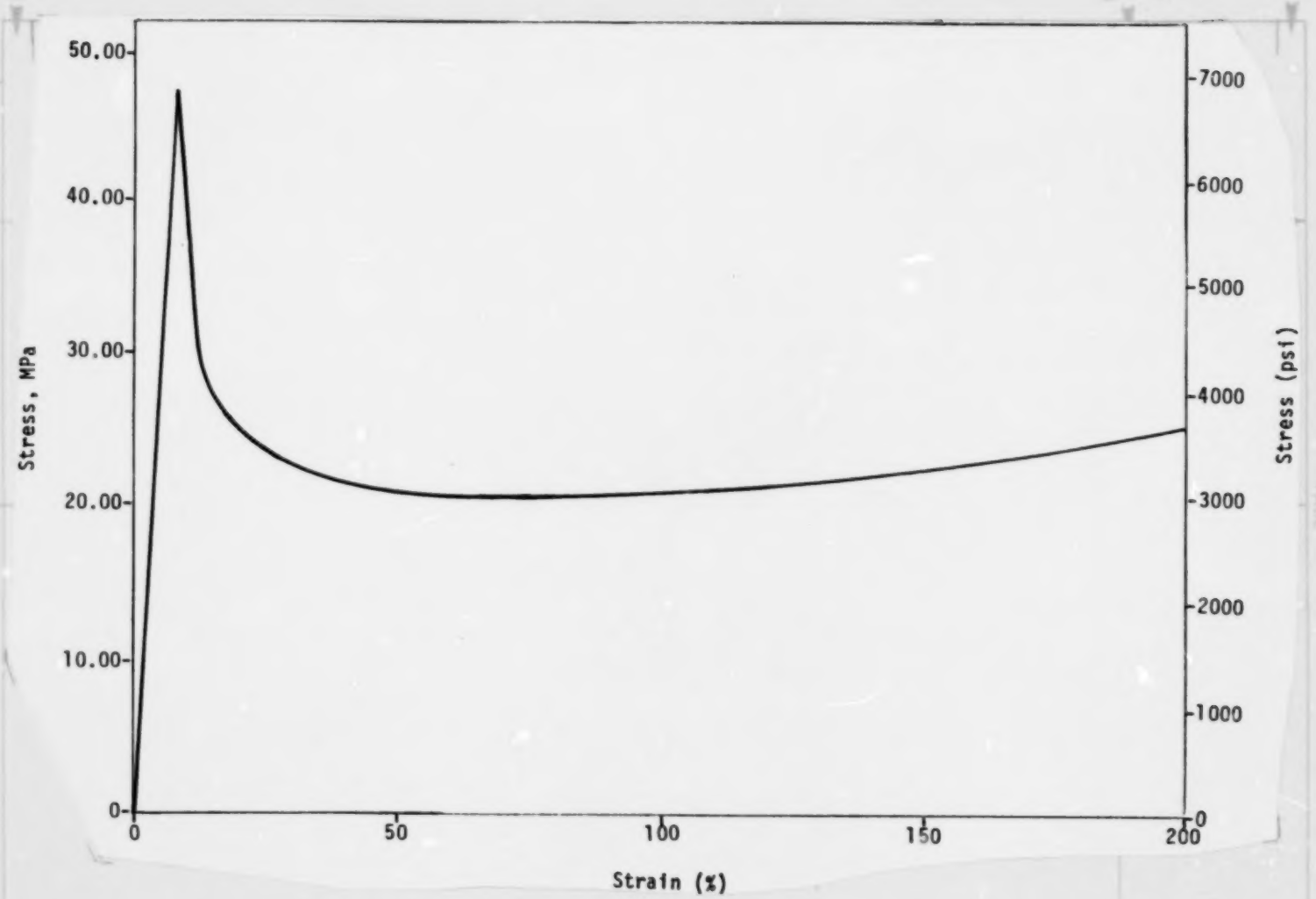

Figure 1. Stress-strain curve for reinforced R-960.

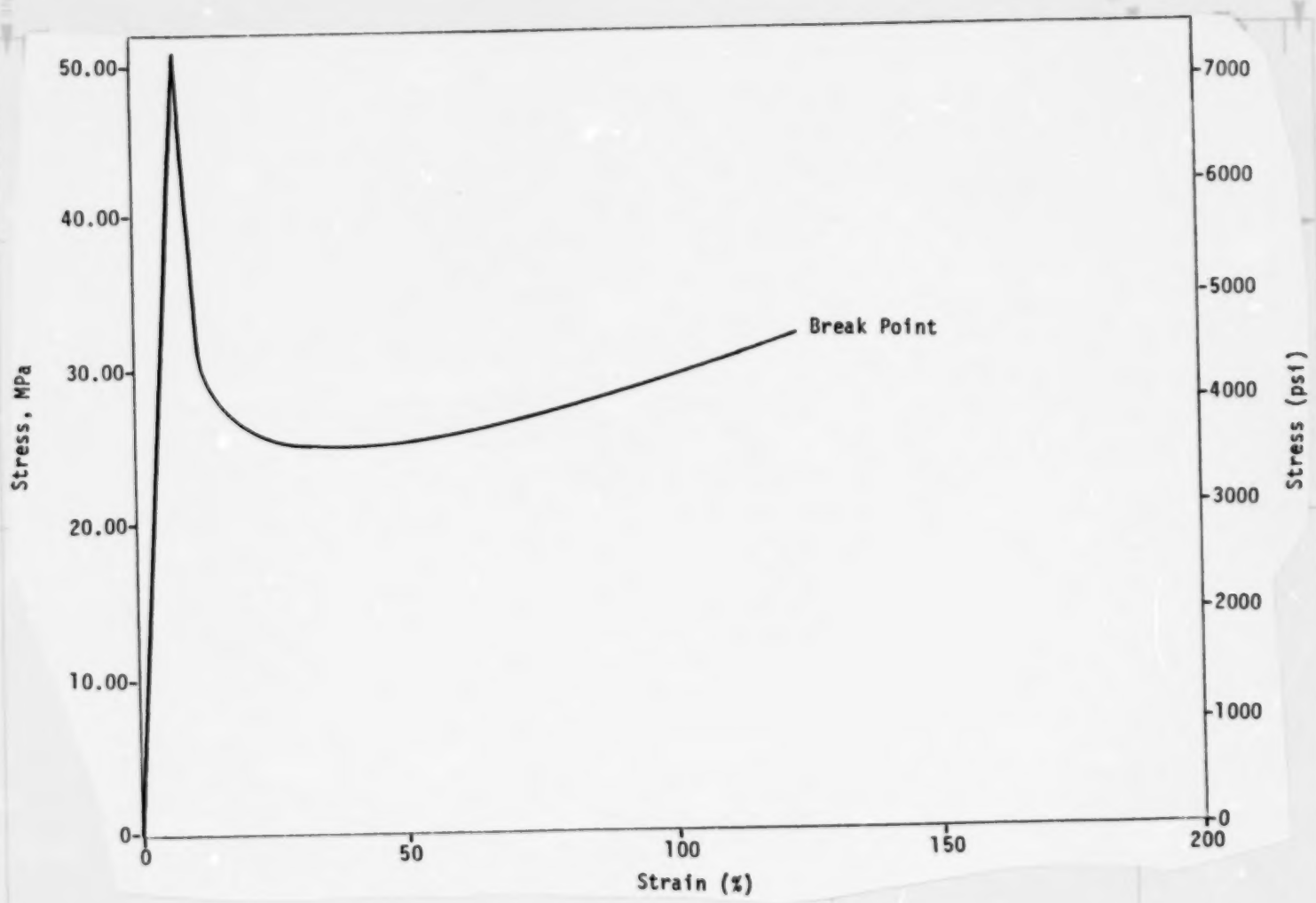

Figure 2. Stress-strain curve for reinforced R-960/CX-100.

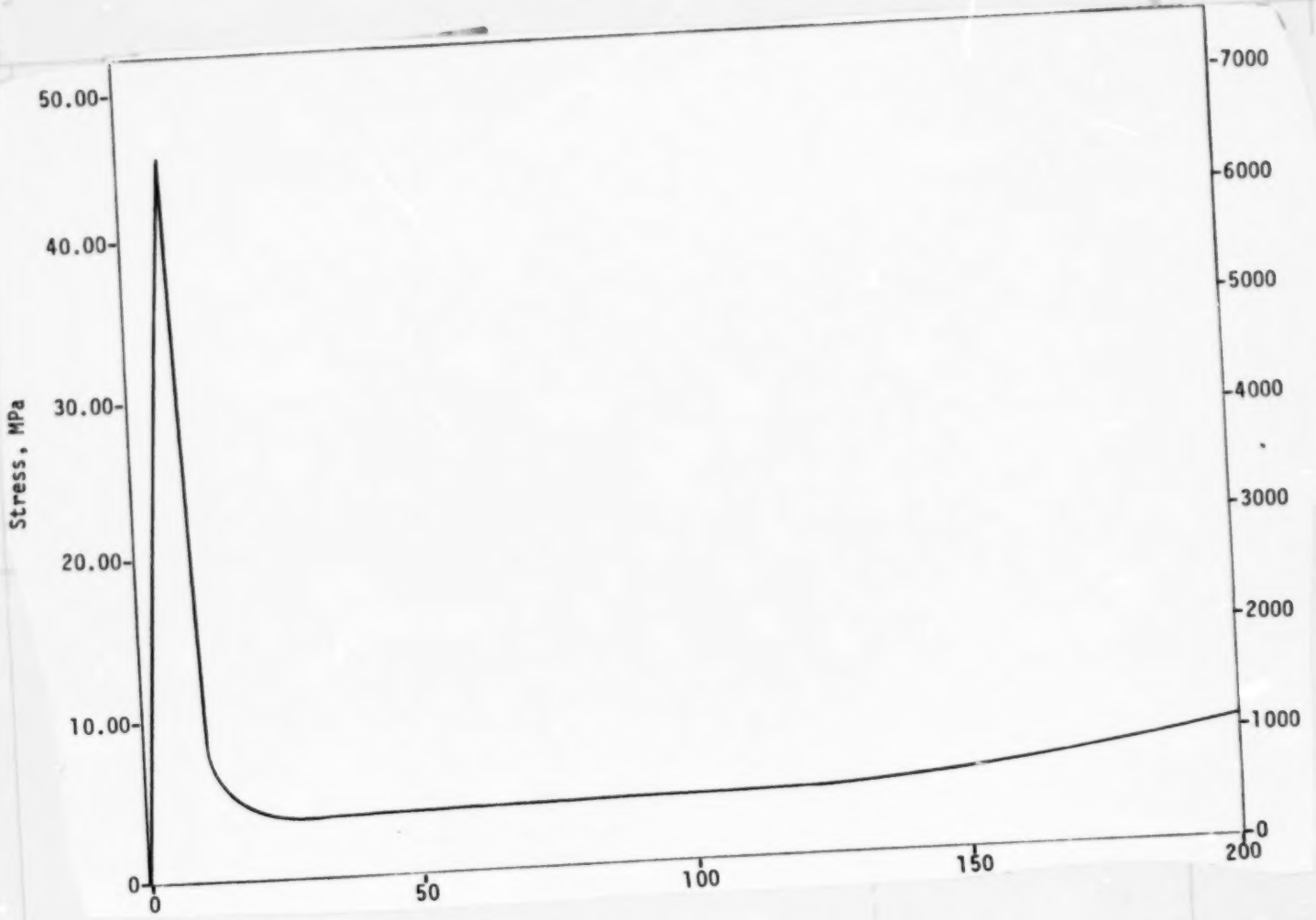

Figure 3. Stress-strain curve for reinforced R-962.

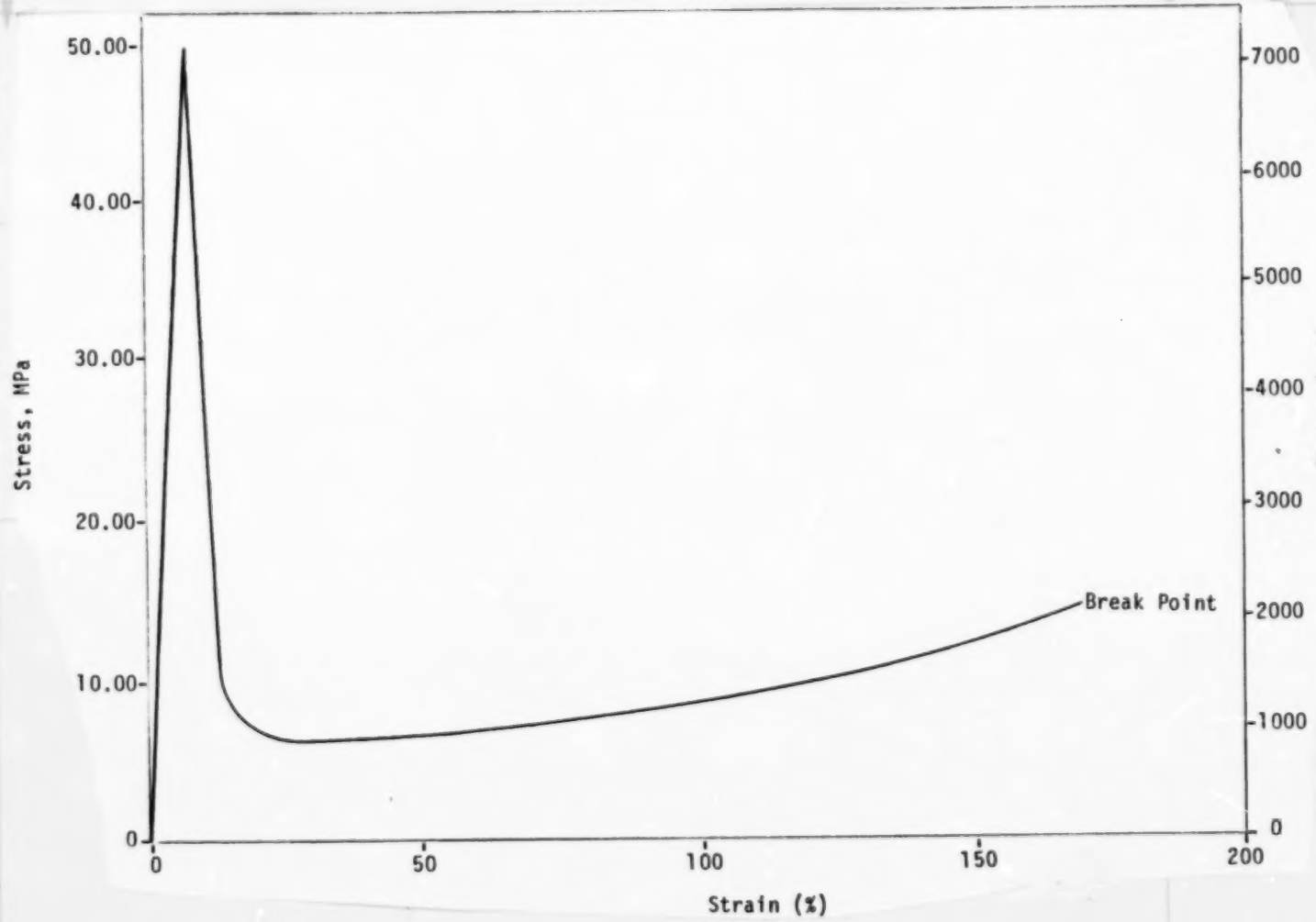

Figure 4. Stress-strain curve for reinforced R-962/CX-100.

TABLE 2. TENSILE PROPERTIES OF COATINGS SUPPORTED ON FIBERGLASS REINFORCEMENTS

Properties \ Specimens	R-960	R-960/CX-100	R-962	R-962/CX-100
Elastic Modulus in Tension, MPa (psi)	610 ± 34 (88000 ± 5000)	650 ± 24 (94000 ± 3500)	590 ± 34 (85000 ± 5000)	660 ± 28 (95000 ± 4000)
Yield Stress, MPa (psi)	48.6 ± 1.4 (7040 ± 210)	51.2 ± 2.1 (7430 ± 300)	46.9 ± 2.8 (6800 ± 410)	51.1 ± 2.3 (7410 ± 330)
Strain @ Yield, %	8.0 ± 0.5	7.9 ± 0.9	8.0 ± 0.6	7.9 ± 0.9
Stress @ Transfer, MPa (psi)	21.4 ± 1.0 (3100 ± 150)	24.1 ± 2.8 (3500 ± 400)	3.6 ± 0.6 (520 ± 80)	6.8 ± 0.6 (990 ± 90)
Stress @ Break, MPa (psi)	26.2 ± 1.7 (3800 ± 240)	30.3 ± 1.6 (4400 ± 230)	10.3 ± 1.4 (1500 ± 200)	14.1 ± 1.9 (2040 ± 280)
Strain @ Break, %	260 ± 25	120 ± 10	530 ± 10	170 ± 30

Figures 5 through 8 are stress-strain graphs of the unsupported coatings. The absence of the graphical discontinuity (~3% strain) corresponding to casing failure is now replaced with the yield points of each coating. Table 3 presents the quantitative data characterizing the above.

Review of Figures 5 through 8 and Table 3 suggests the same behavioral trends for unsupported coatings as observed for reinforced specimens. In addition, comparison of Tables 2 and 3 indicates the jacket formation should not significantly alter the expected properties of the coatings for encapsulation applications.

Hardness Tests

Shore A and pencil hardness tests were conducted to characterize further the relative hardnesses of the coatings. Tests were conducted in accordance with ASTM D-2240 and MIL spec C-832868 (USAF), Amendment 1, respectively (see Appendix A for details).

Table 4 summarizes these tests. R960/CX-100 was clearly the hardest coating.

Table 5 compares properties obtained in these studies with the vendor's published data. This illustrates the retention of anticipated high properties during jacket formation.

Immersion Tests

A final screening test involved immersion of coatings in deionized water. This test followed the guidelines of ASTM-D-1239 (Appendix A), whereby neat coatings of R-960 and R-960/CX-100 were prepared. Table 6 lists the results of this study. It is apparent that swelling and imbibing of water occurred in the R-960 coating, but much better resistance was observed for the R-960/CX-100 specimens (characteristic of a cross-linked system's enhanced chemical resistance[4]).

Table 7 lists the vendor's comparison of properties of R-960 with R-960/CX-100. The scoring system (10 = best, 1 = worst) allows relative comparison and has no other numerical correlation.

SELECTED ATMOSPHERIC-TEMPERATURE-CURING COATING

The strong mechanical and chemical properties, general processability, and workability lead to the selection of Neorez R-960 cross-linked with CX-100 as the coating for detailed studies. These studies are described in the following sections.

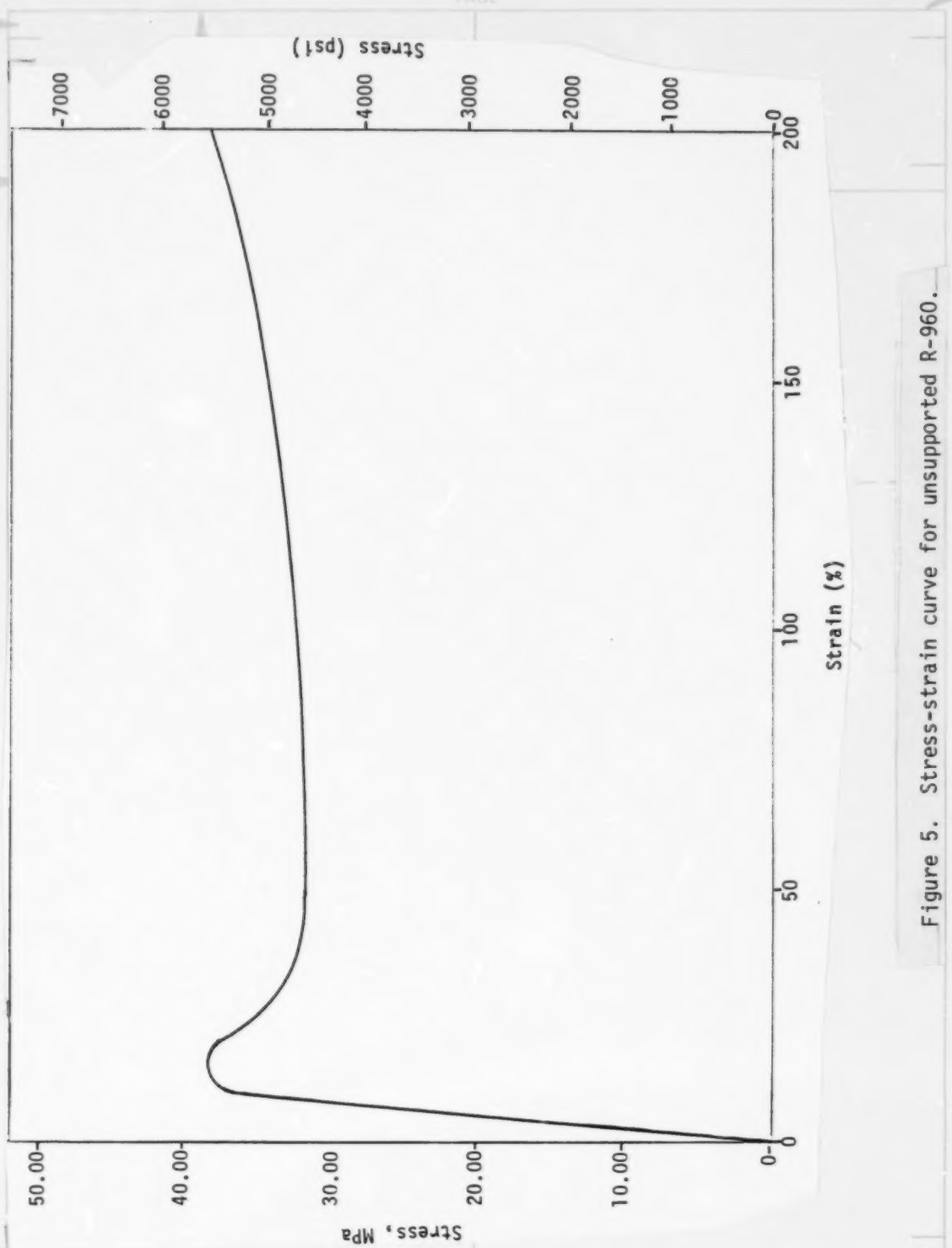

Figure 5. Stress-strain curve for unsupported R-960.

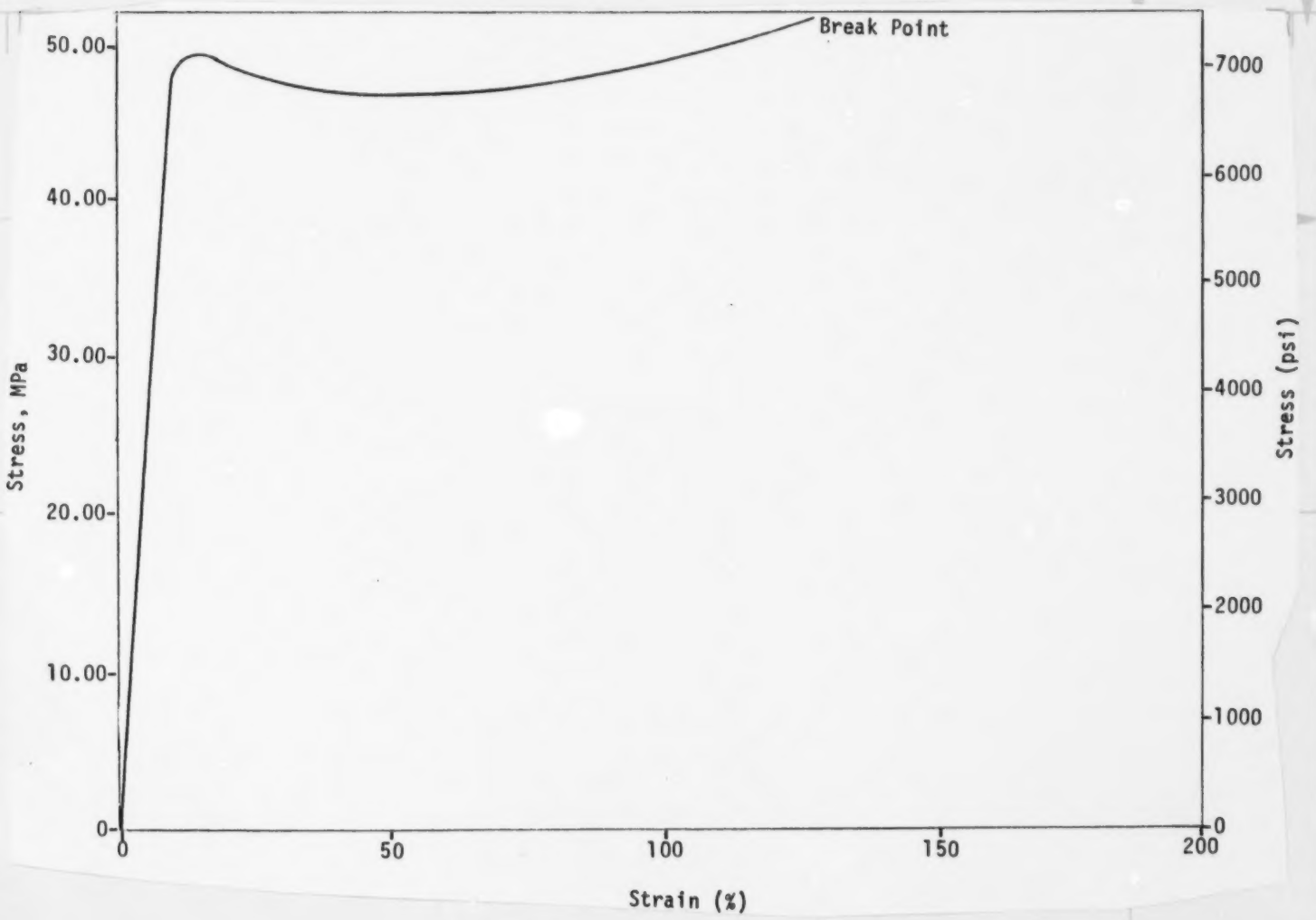

Figure 6. Stress-strain curve for unsupported R-960/CX-100.

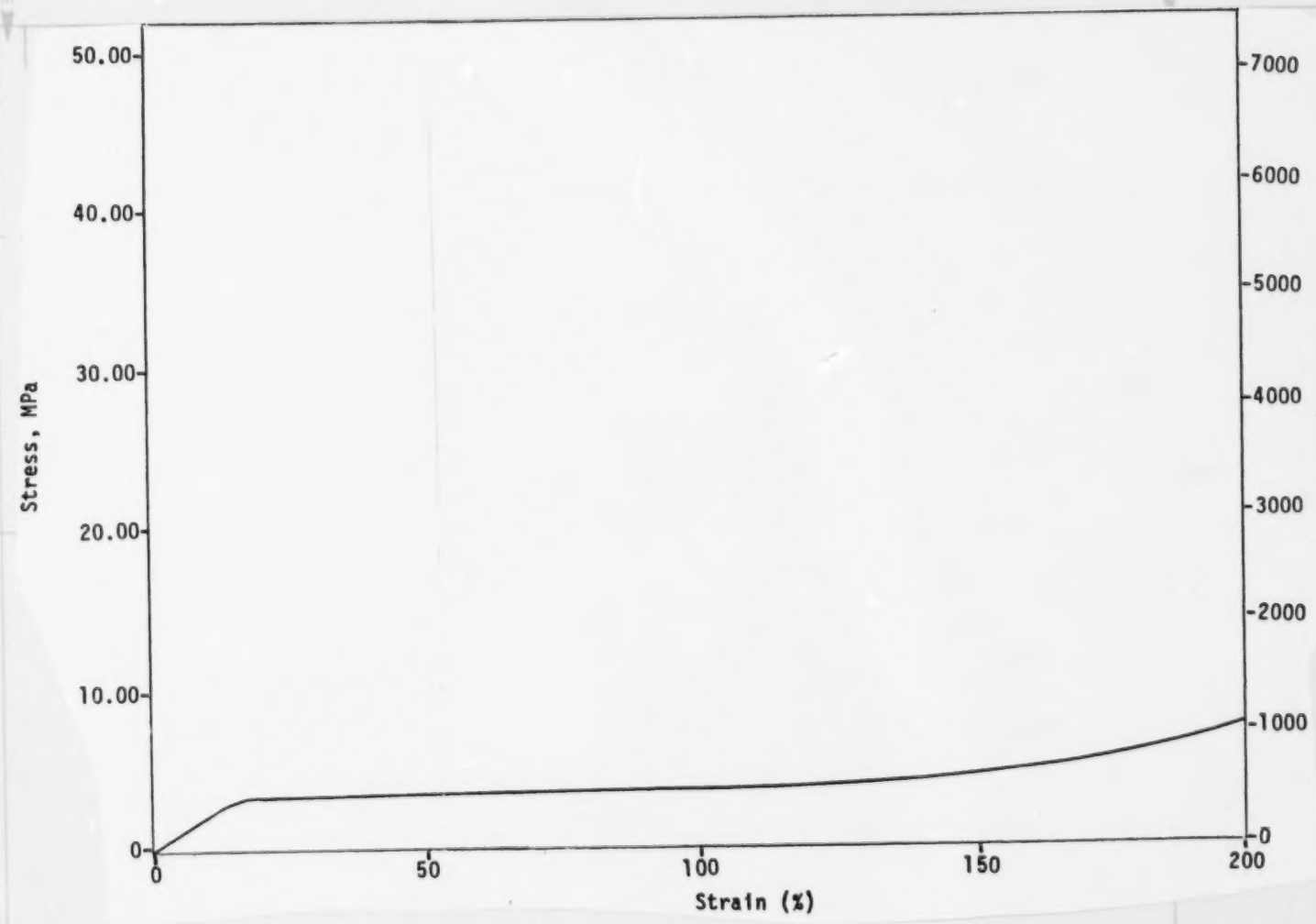

Figure 7. Stress-strain curve for unsupported R-962.

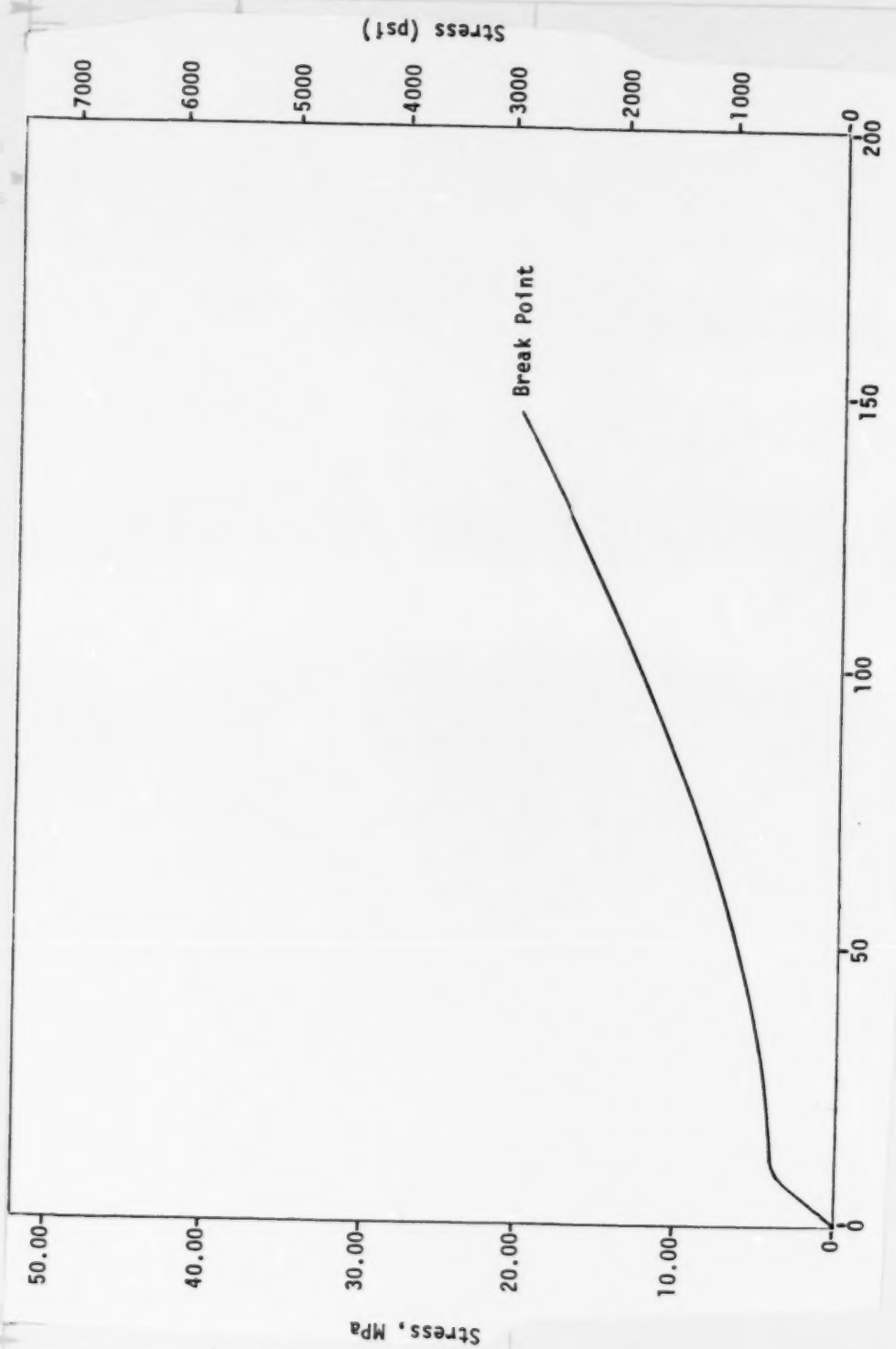

Figure 8. Stress-strain curve for unsupported R-962/CX-100.

TABLE 3. TENSILE PROPERTIES OF UNSUPPORTED COATINGS

Property \ Specimens	R-960	R-962/CX-100	R-962	R-962/CX-100
Elastic Modulus in Tension, MPa (psi)	393 ± 52 (57000 ± 7500)	490 ± 41 (71000 ± 6000)	21.4 ± 2.8 (3100 ± 400)	43.4 ± 2.1 (6300 ± 300)
Yield Stress, Mpa (psi)	38.6 ± 3.1 (5600 ± 450)	48.3 ± 2.1 (7000 ± 300)	4.1 ± 0.3 (600 ± 50)	4.5 ± 0.5 (650 ± 70)
Strain @ Yield, %	14 ± 2	13 ± 2	19 ± 5	16 ± 6
Stress @ Break, MPa (psi)	40.0 ± 2.8 (5800 ± 400)	49.7 ± 1.5 (7200 ± 220)	10.3 ± 2.1 (1500 ± 300)	21.4 ± 3.1 (3100 ± 450)
Strain @ Break, %	220 ± 15	130 ± 5	580 ± 30	150 ± 11

TABLE 4. HARDNESS TESTS

Property \ Specimen	960	960/CX-100	962	962/CX-100
Hardness (Shore A)	55	61	NR*	20
Hardness (Pencil)	4H	8H	H	3H

*No response on durometer upon testing

TABLE 5. COMPARISON WITH VENDOR'S DATA OF PROPERTIES OF COATING SPECIMENS WITH AND WITHOUT REINFORCEMENT

Property \ Specimen	Reinforced Coating				Neat Coating				Vendor[1]			
	R-960	R-960/CX-100	R-962	R-962/CX-100	R-960	R-960/CX-100	R-962	R-962/CX-100	R-960	R-960/CX-100	R-962	R-962/CX-100
Yield Stress, MPa (psi)	48.6 ± 1.4 (7040 ± 210)	51.2 ± 2.1 (7430 ± 300)	46.9 ± 2.8 (6800 ± 410)	51.1 ± 2.3 (7410 ± 330)	38.6 ± 3.1 (5600 ± 330)	48.3 ± 2.1 (7000 ± 300)	4.1 ± 0.3 (600 ± 50)	4.5 ± 0.5 (650 ± 70)	34.5 (5000)	DNP[2]	DNP	DNP
Strain @ Break, %	260 ± 25	120 ± 10	530 ± 20	170 ± 30	220 ± 15	130 ± 5	580 ± 3	150 ± 11	170	DNP	600	DNP
Hardness (Pencil)	4H	8H	H	3H	4H	8H	H	3H	4H	7H	DNP	DNP
Hardness (Shore A)	55	61	NR[3]	20	53	60	NR	19	-	-	-	-
(Sward)	-	-	-	-	-	-	-	-	44	44	8	DNP

(1) Polyvinyl Chemical Industries
(2) DNP - Data not provided by Vendor
(3) NR - No response on durometer upon testing

TABLE 6. WEIGHT GAIN (%) DURING IMMERSION TESTS

Sample \ Immersion Time (hrs)	4	24	48	72	264	Final observations
R-960	32.5	72.3	90.1	98.4	105	Embrittled coating was curled, visually swollen, hazy, opaque white (originally amber and transparent)
R-960/CX-100	0.97	1.83	2.05	3.22	5.37	No visual swelling, a very slight haze on corner of one specimen

TABLE 7. VENDOR'S COMPARISON OF R-960 VERSUS CROSS-LINKED R-960

Film Tests	Test Results for R-960	Test Results for R-960/CX-100
Dry time, min. to tack-free 75°F 50% R.H.	210	210
Sward Hardness		
24 hr	38	38
7 days	44	44
Taber Abrasion CS-17, 1000 g/1000 cycles mg	28	15
Relative Comparison of Films	**Score***	**Score***
Chemical resistance, 24 hr *		
IPA	1	10
Toluene	10	10
IN KOH	10	10
Mar resistance		
24 hr	6	9
1 week	6	9
Gardner scrub resistance		
500 cycles 2% Spic & Span	10	-
500 cycles D.I. Water	9	-

*Vendor's scoring system (10 = best, 1 = worst) allows relative comparison and has no other numerical correlation.

Note: The addition of cross-linker CX-100 to R-960 results in films that are comparable to a moisture cured urethane which is accepted for abrasion and chemical resistant coatings. This comparison indicates the high level of performance that can be attained with R-960/CX-100.

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SECTION 5

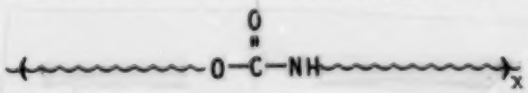
CHARACTERIZATIONS OF COATING

This section describes the chemistry, formulation, and a probable mode of coating formation that aids to characterize the coating. This work provided a baseline that led to the coating evaluation studies in Section 6.

CHEMICAL NATURE OF COATING

Because of the patent protection being sought by the vendor, exact chemical structure cannot be disclosed; however, a general discussion is included to describe better the features of the coating and the processing development conducted in the laboratory.

Linear, aliphatic polyurethanes are of the general structure



with the aliphatic portions of the polymer incorporated into the backbone.

The development of R-960, a stable, waterborne, colloidal (<0.1 micron) dispersion of polyurethane latices is an attractive feature as industry moves away from solvent-based systems. Dispersion is accomplished by incorporating carboxyl functional groups (COOH) along the backbone structure. The concentration of these groups, when charged in basic solutions, aids solubilizing the polymer in water.

Cross-linking of polymer chains enhances the final coating's chemical resistance and mechanical toughness.[5,6] Cross-linking, at atmospheric temperatures, is accomplished using polyfunctional, 100% active aziridines which react with the carboxyl functionalities of the dispersion.

ADVANCED COATING FORMULATION

Four candidate resins were identified and subsequently tested (see Section 4), from which a single formulation was advanced. The formulation utilized for detailed studies is identified in Table 8. This formulation was developed during the later stages of the selection process.

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TABLE 8. ADVANCED FORMULATION

Trade name	Content (parts by weight)
Neorez R-960	100.0
Cross-linker CX-100	3.0
Deionized Water	3.0
Foamaster W-55	0.04

Table 9 lists additional data characterizing components of the formulation.

The method of mixing resin components is a critical process affecting the quality of the finished coating. Improper mixing leads to foaming and/or heterogeneous areas in the cured coating that may diminish both mechanical properties and its performance as a sealant.

In the laboratory, formulation was accomplished in the apparatus sketched in Figure 9. The mixing method was as follows:

> Neorez R-960 was placed in a resin flask and the appropriate amount of Foamaster W-55 was added. The flask was fitted with a condenser for volatiles, a variable-speed mechanical stirrer, and a calibrated funnel holding cross-linker CX-100 diluted ½ with deionized water. (Undiluted cross-linker was difficult to mix into solution and led to the premature gelation of polymer during stirring. The resultant coatings had reduced mechanical properties.) CX-100 was added at a rate of 1 ml/min while the solution was stirred at 80 to 100 rpm. (During stirring, care must be taken to prevent foaming which can remain in the formulated resin and appear as voids in the cured coating. However, if foam forms, removal is hastened with additional Foamaster W-55 at 0.01 pbw. This solution is left free-standing until the bubbles disappear.) Mixing of formulated resin was routinely continued for 30 minutes after completion of cross-linker addition.

Formulated resin was stored for up to 3 days in sealed containers. If used after 3 days, the resin was re-inoculated with CX-100 without loss of mechanical properties.

All components of the formulation have adequate shelf lives for storage for at least 1 year. (Neorez R-960 should be mixed before use.)

COATING FORMATION

The mode of coating formation is presumed to occur in the following manner which is similar to the mechanism described by Grief.[7] Upon application of the formulated resin to an encapsulate's surface (described in Section 7), the curing process is believed to commence as water evaporates. During this period, the dispersed colloidal resin particles approach each other. As water continues to evaporate, the particles make contact and flow together with the aid of a coalescing solvent. (The solvent remains because it boils at a higher temperature than water.) The solvent-rich system now formed is analogous to conventional solvent-lacquer systems. As coalescing solvent evaporates, the particulate nature of the resin vanishes. Cross-linking is initiated in solution and accelerates with evaporation of water and solvent. Crystallization of polymer occurs primarily in these latter stages of cure. A schematic of the curing process is given in Figure 10.

Although the kinetics of coating formation are not clear, in the laboratory it was found that the overriding rate-determining processing step was water evaporation. This regulated both the curing period and the time lapse required between applying multiple coatings.

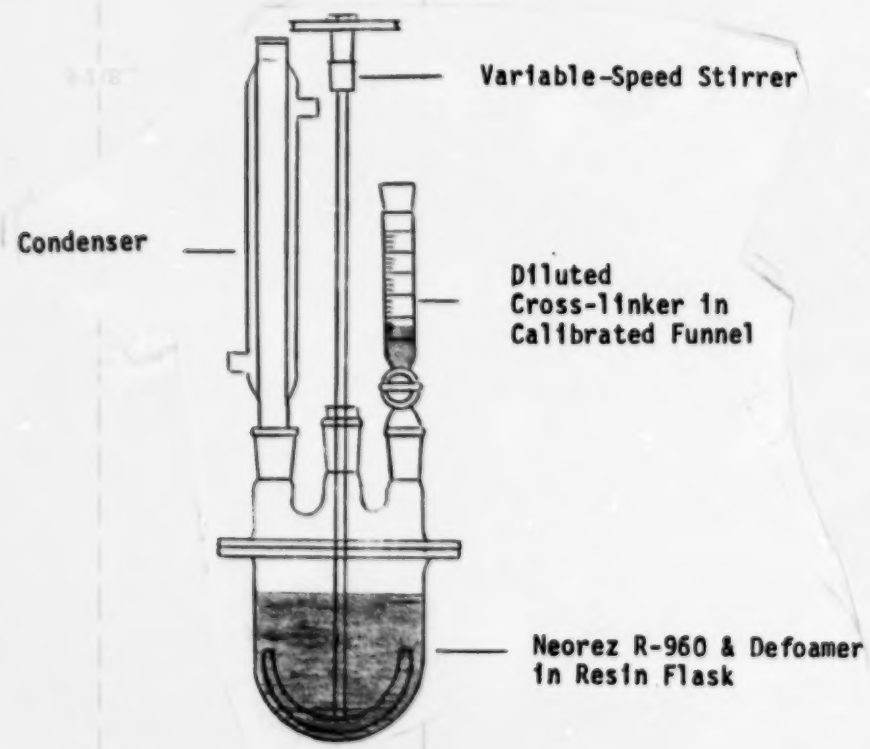

Figure 9. Sketch of formulation apparatus.

TABLE 9. ADDITIONAL VENDOR'S DATA

Description / Tradename	Chemical Type	Solvent	Viscosity Pa·s (cps)	pH	Solids (%)	Density g/cm³ (lb/gal)	Vendor
Neorez R-960	Aqueous Colloidal Dispersion of an Aliphatic Urethane	Water	0.4 - 0.8 (400 - 800)	7.5 - 8.5	34	1.05 (8.8)	Polyvinyl Chemical Industries
Cross-linker CX-100	Polyfunctional aziridine (100% active) liquid	None	0.2 (200)	4.5	-	1.07 (8.9)	Polyvinyl Chemical Industries
Foamaster W-55	Surfactant (100% active)	-	-	5.0 - 5.5	-	0.917 (7.65)	Diamond - Shamrock

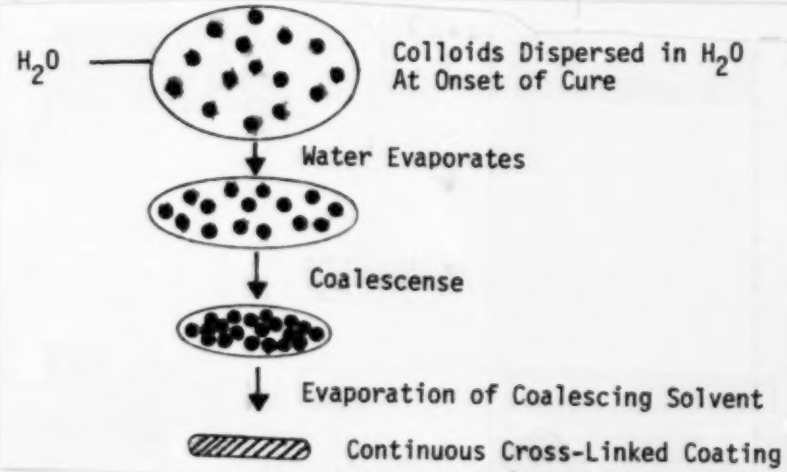

Figure 10. Coating formation.

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SECTION 6

COATING EVALUATIONS

Evaluation of the coating was advanced by curing studies. These studies gave some insight as to expected engineering properties under simulated service conditions. In addition, a valuable quality assurance criterion was identified.

COATING CURING STUDIES

The formation of a coating possessing engineering properties suitable for encapsulation applications required knowledge of its curing behavior. Improperly cured coatings subjected to handling, transportation, or deposition might fail.

Hardness studies were conducted to monitor the degree of cure. Hardness generally correlates with mechanical strength and rigidity[8] and it is important for industrial coatings applications because it can indicate completion of polymerization and solvent evaporation until hard-dry.[9]

Studies were conducted in accordance with ASTM D-2240 (Shore A) and military specification MIL-C-83286B (USAF), amendment 1 (pencil hardness) (see Appendix A).

Figure 11 shows a hardness-time study of the coating when subjected to diverse operating conditions that represent colder (10.0°C /50°F/), average (23.9°C /75°F/), and desert (48.9°C /120°F/) temperatures. It is evident that within the first few days, the hardness of the coating increases at a rapid rate. This rapid development in green strength is an essential feature for industrial applications.[10] After about 5 to 7 days, curing continues at slower but still appreciable rates.

At 48.9°C (120°F), the coating was essentially at complete cure within ~7 days. The 23.9°C (75°F) and 10.0°C (50°F) specimens were 60% and 40% of this hardness value, respectively.

Specimens for the study were 12.7 mm (0.25 in.) thick and were formed by casting formulated resin into aluminum dishes. This thick element was required for testing and does not represent the thicknesses obtained on encapsulates prepared in the laboratory (see Section 8). However, the study does aid in characterizing the behavior of the coating in diverse atmospheric temperatures and its relative hardness during cure.

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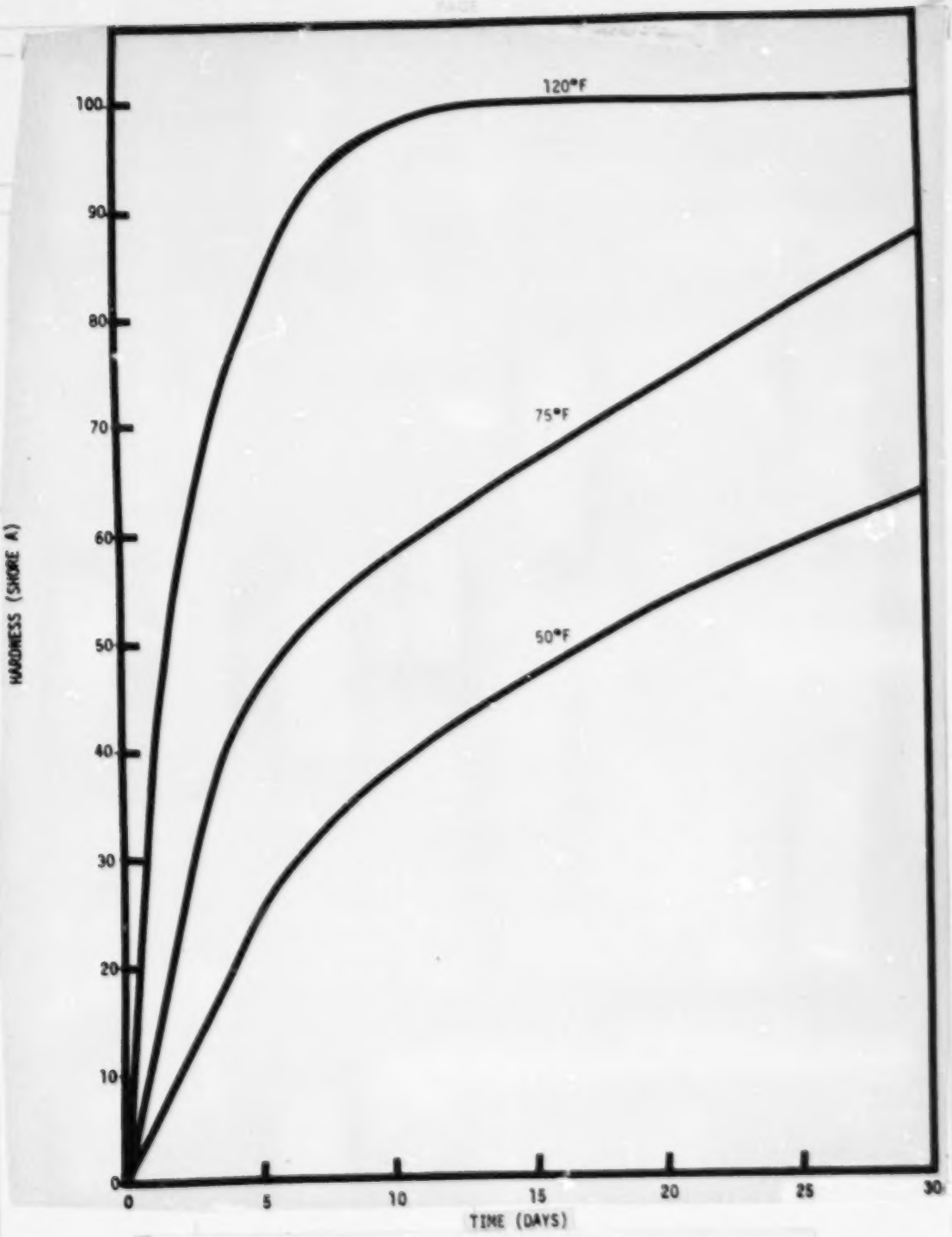

Figure 11. Hardness-time study for coating at cold, average, and desert temperatures.

Testing scratch resistance of the coating's surface was evaluated using pencil hardness tests. Table 10 lists results. Specimens were single layers of coating ~0.4 mm (0.016 in.) thick on aluminum plates. (This thickness per coat is similar to those achieved during fabrication.) A reduction in surface hardness is evident for coatings cured at lower temperatures.

HARDNESS/TENSILE STRENGTH RELATIONSHIP

During the resin selection procedure and curing studies, an empirical relationship between hardness and tensile strength was observed that might serve as a model for developing quality assurance methods for the field. The relationship is supported by the reinforced plastic industry's technique for determining the degree of cure of a resin, from a green state to a fully cured state, by hardness measurements, allowing the selection or rejection of parts.[11]

Figure 12 shows a correlation between hardness and strength for R-960/CX-100 specimens. The rapid buildup of strength at hardnesses above 60 Shore A units might indicate the lower limits of hardness desirable before handling, transportation, or deposition of encapsulates.

TABLE 10. SCRATCH RESISTANCE OF COATING

Curing Time, hr \ Cure Temp., °C (°F)	10° (50°)	24° (75°)	49° (120°)	Vendor's Data 23.9° (75°)*	Vendor's Data 93.3° (200°)**
4	6B	7H	8H	7H	7H
24	6B-5B	8H	8H	-	-
48	-	8H	8H	-	-
72	3B	8H	8H	-	-
96	2B	8H	8H	-	-
120	H	8H	8H	-	-
240	3H	8H	8H	-	-

*4 hours defined by vendor as complete cure schedule @ 75°F.

** Cured in air oven @ 200°F for 10 minutes.

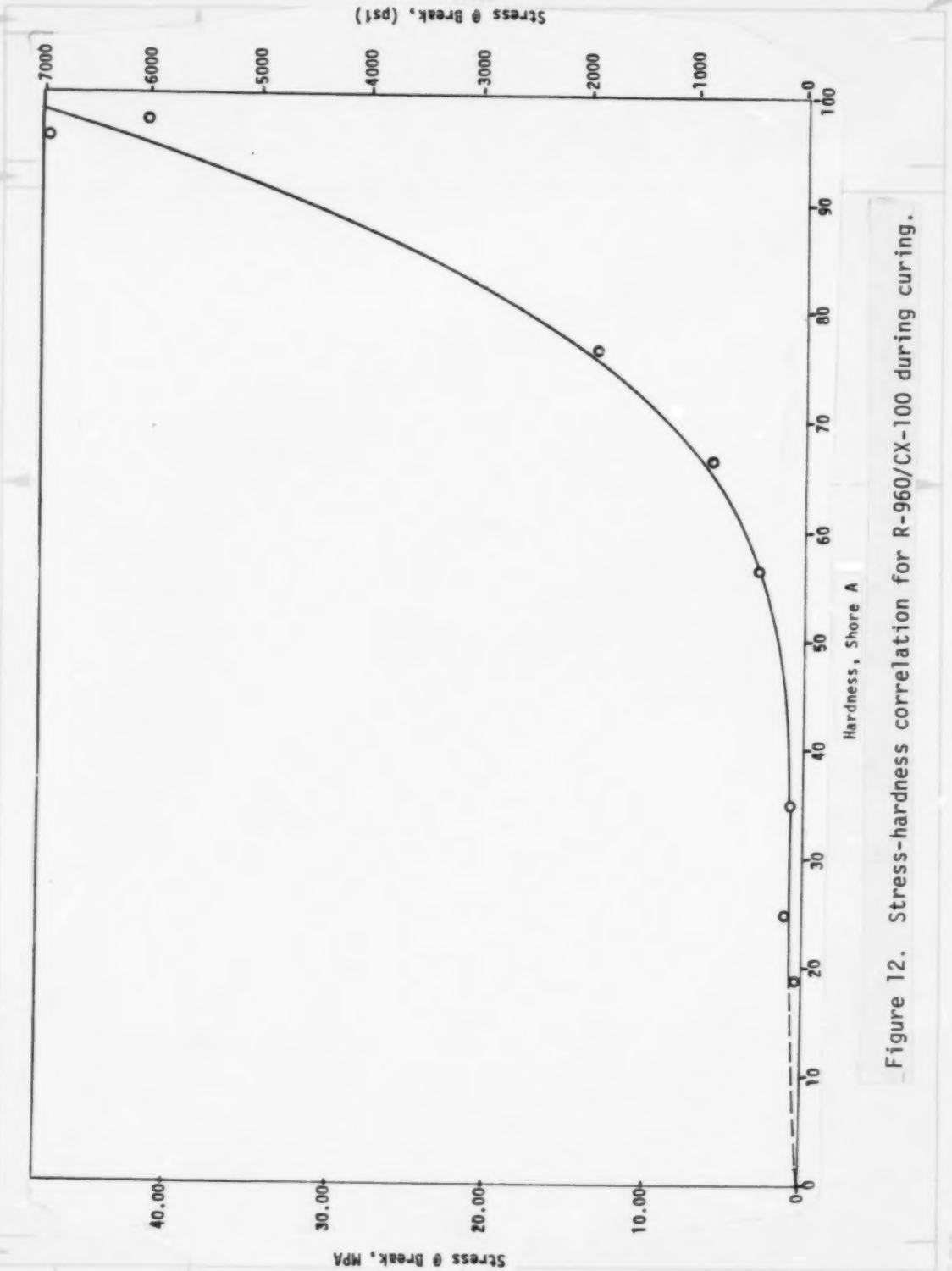

Figure 12. Stress-hardness correlation for R-960/CX-100 during curing.

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SECTION 7

METHOD OF ENCAPSULATION

The purpose of this phase of the work was (1) to establish a method of encapsulation suitable as a basis for large-scale production, and (2) to fabricate specimens for testing performance capabilities of encapsulates.

Test specimens were fabricated as cylinders measuring 63.5 mm (2.50 in.) in diameter by 88.9 mm (3.50 in.) high. Specimen structures were characterized by a seam-free, flexible outer resinous coating bonded to a stiff, load-bearing inner reinforcement, with the free inner volume accommodating the hazardous waste. Seam-free jackets were readily achieved according to the method described below. Tenacious adhesion of coating to reinforcement occurred as a result of an incremental penetration of the resin, allowing the formation of both mechanical locks and chemical bonds. Although chemical adhesion between coating and reinforcement was deemed to have occurred, assessment of respective contributions of mechanical locks and chemical bonds to overall adhesive bond strength was not possible because of the nature of the adherents.

In the laboratory, fabrication of encapsulates proved to be a simple procedure. The resin coatings were applied by brush, and multi-layered coatings were inseparable and homogeneous.

FABRICATION OF TEST SPECIMENS

Figure 13 shows a fiberglass encasement (containing NaCl) placed in a polyethylene mold during fabrication. Use of PE mold requires no auxiliary releasing agents, thereby facilitating demolding operations. This feature is particularly attractive because no residuals remain on the encasement's surfaces (contrary to externally applied releasing agents) that could inhibit coating-reinforcement adhesion. The successful use of PE is consistent with the anticipated implementation of low-cost PE molds for spray-up operations in the field.

A fiberglass encasement holding table salt is shown in Figure 14. The walls of this encasement serve as the substrate for application of the atmospheric-temperature-curing resin.

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Atmospheric curing resins were applied to fiberglass casings as illustrated in Figure 15. In the laboratory, the resin was brushed on and allowed to dry, the encapsulate was turned over, and the uncoated area was treated, thus completing the jacketing operation. A 208-liter (55-gal) container pretreated with fiberglass spray-on might be jacketed with atmospheric-temperature-curing resins by a commonly employed spray-on technique. Hardware stores feature a spray-on apparatus that employs paint direcly from 18.9-liter (5-gal) containers. In the field, management of 208-liter (55-gal) drums might also utilize resins directly from the containers in which the formulation was carried out. Figure 16 shows the finished test specimen.

PIGMENTATION OF COATING

In the following section, modifications of the coating formulation are described using two pigmentation techniques. The use of pigmentation was a clear advantage for high quality encapsulation, although good specimens were prepared primarily without pigmented coatings.

A key processing problem is recognizable in Figure 17. The coating is amber and transparent, which hinders visual detection of uncoated areas when applied to the substrate. Complete wetting of all surfaces of the reinforcement is essential for proper sealing.

Figure 13. Fiberglass encasement containing salt in PE mold.

Figure 14. Fiberglass-encased salt.

Figure 15. Atmospheric-temperature-curing resin jacketing operation.

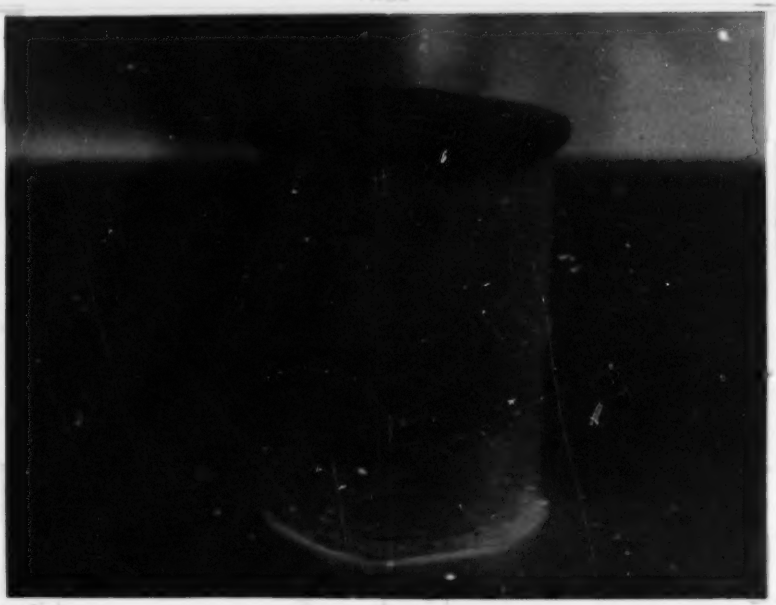

Figure 16. Completed atmospheric-temperature-curing resin test specimen.

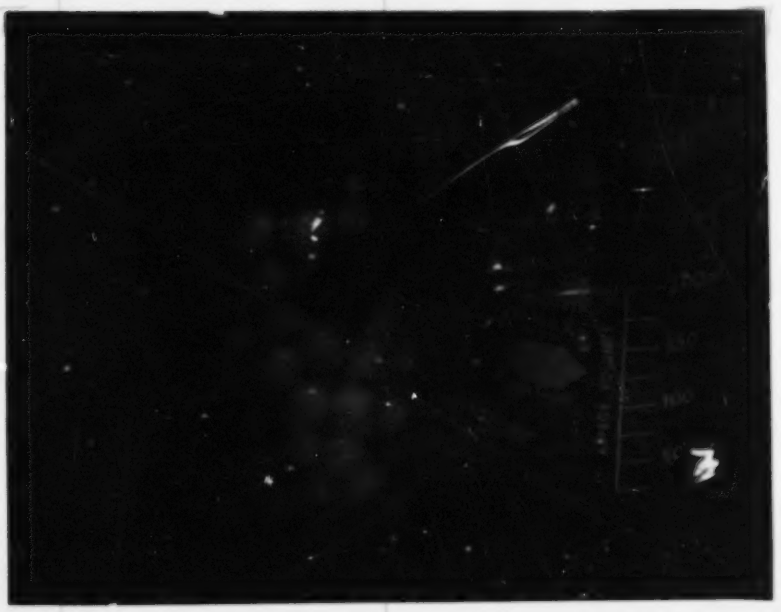

Figure 17. Jacketing without pigmented resin (plastic micro-balloons represent small containers of waste.)

Two pigmentation systems have been identified to mitigate the above difficulty. Figure 18 shows the coating pigmented with an organic dye (bromocresol green), neat coating, and colored with TiO_2 (from left to right, respectively). Both pigmentation systems are water dispersible and compatible with the resin system.

Figure 19 shows an encapsulate being coated with the dye-pigmented coating. The visual contrast enhancing sealing is evident.

The predispersing of organic pigmentation into the coating was accomplished by preparing an aqueous solution of ~20 pbw of sodium salt of the organic dye. Addition of this solution was made until a deep blue color was obtained (<0.1 pbw).

TiO_2 was initially coated with a nonionic surfactant (Dispersant NI-W, Chevron Chem. Co.) and dispersed in water as a slurry. This slurry was added to the coating (<0.1 pbw).

Although two pigmentation systems were identified, the organic dye was utilized for the production of a few specimens because it was easier to disperse and subsequently process the coating.

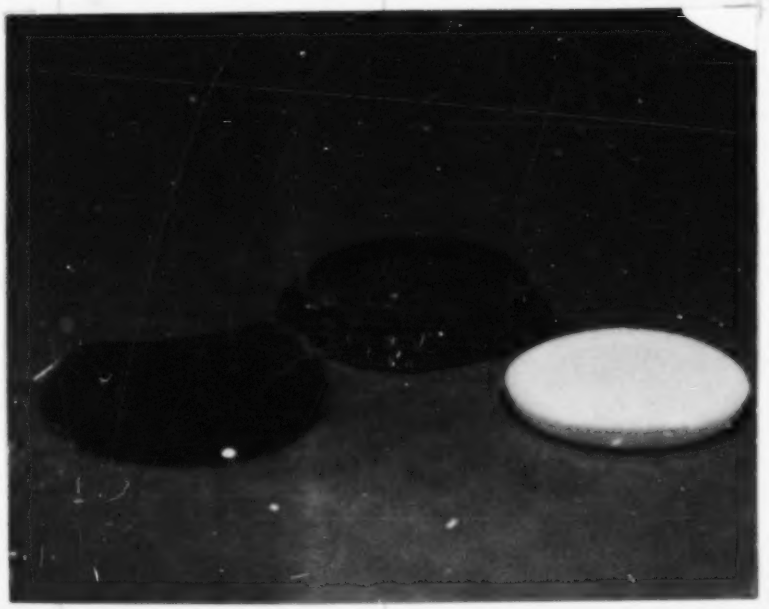

Figure 18. Pigmented coatings: Organic dye, neat coating, TiO_2, respectively.

Figure 19. Application of dye-pigmented coating.

SECTION 8

PROCESS EVALUATIONS

Encapsulates were tested to determine their response to mechanical and chemical stresses that they may encounter during manipulation, transportation, and deposition. Emphasis was placed on verification of contaminant localization capabilities. Similar attention was directed at achieving coatings with adequate thicknesses to assure high mechanical performance. (This high performance was considered necessary to meet the stringent DOT requirements imposed on hazardous waste containers.)

COMPRESSION TESTS

The nature of encapsulates under compressive stresses was investigated with respect to mechanical loads applied to specimens in vertical positions. The extent of compression in which encapsulates maintained functional integrity* was characterized by jacket thickness. Since the fiberglass reinforcement's thickness was essentially fixed, the increases in jacket thickness reported here were due to variance in outer polyurethane coating.

Generally, encapsulates with relatively thinner coatings <0.66 mm (<0.026 in.) failed early in compression, many along with the reinforcement's failure. Thicker coatings >2.97 mm (>0.117 in.) performed much better, a few achieving ~20% deflection before failure.

Figures 20 and 21 show testing equipment and a closer view of a mounted encapsulate, respectively. Compression tests were performed on a Reihle Testing Machine (Model F-S-160) and a Cal Tester (Model TH-5) in accordance with ASTM-C-39-71, but modified to accommodate specimen dimensions (see Appendix A).

To measure the coating thicknesses of specimens to be tested nondestructively, five calibration panels were prepared. Each panel was prepared from a fiberglass base (similar to encapsulate's reinforcement), vertically mounted, and brush-coated as with encapsulates. Measurements of multiple coatings indicated average thicknesses of 0.33 ± 0.008 mm (0.013 ± 0.003 in.) per application. The thickness of each jacket was calculated based on this average thickness and the number of applied coats.

*Functional integrity - based on visual observation that no cracks, crazing, or other flaws occurred on R-960/CX-100 outer coating during compression.

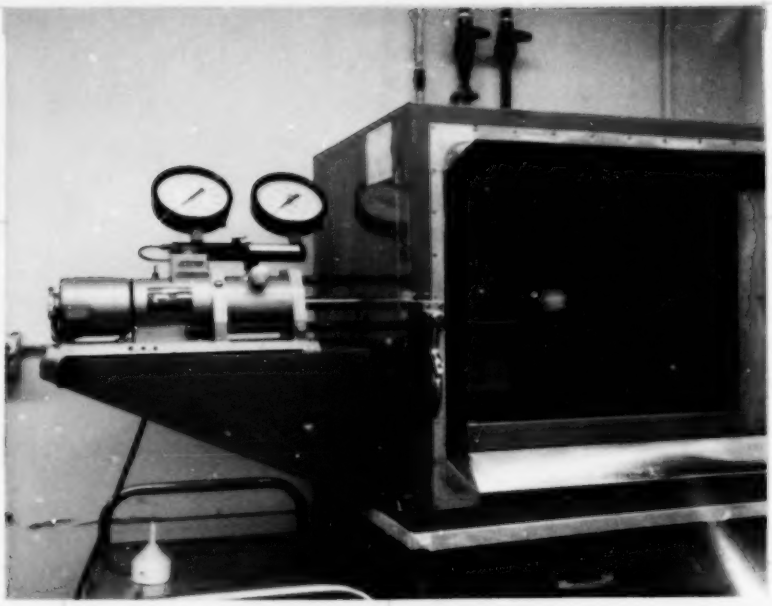

Figure 20. Compression testing equipment.

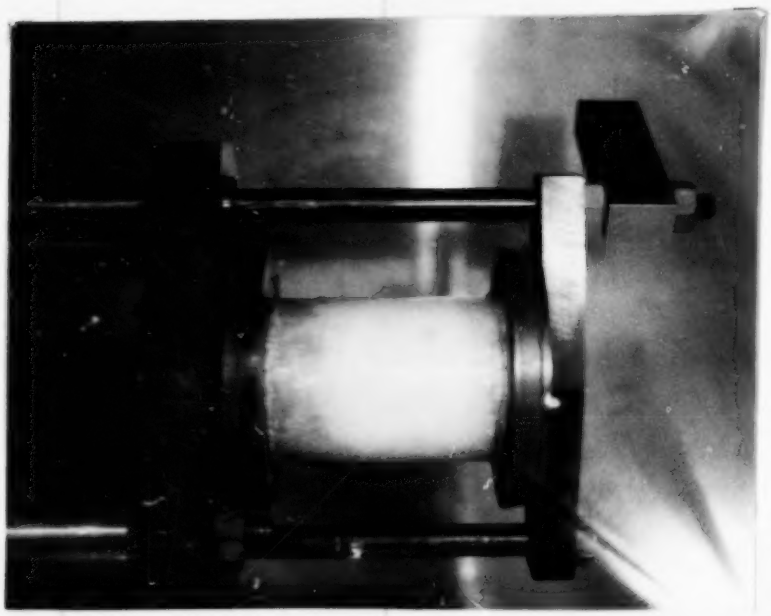

Figure 21. Onset of compression testing.

Failure of encapsulates with relatively thin coatings (<0.66 mm /<0.026 in./) was similar to the performance shown in Figure 22. Here, failures of reinforcement and outer resin were essentially simultaneous, corresponding to ~3% deflection of original specimen size.

Encapsulates with thicker coatings (>1.32 mm />0.052 in./) maintained functional integrity beyond reinforcement failure, as shown in Figure 23.

Coatings with thicknesses of less than ~1.65 mm (~0.065 in.) failed within the first 7% of deflection. Figure 24 illustrates the typical failure mode in which the outer jacket failure occurred along a propagating fracture in the reinforcement.

Compression of specimens with thicknesses in the range of 3.05 mm (0.12 in.) led to satisfactory performance near 20% deflection. Figure 25 shows a specimen that has been compressed and deflected to catastrophic failure (beyond recovery). An important feature is the continuous deformation and drawing of the coating upon stress.

Table 11 summarizes results for compression studies of six specimens containing construction sand. The trend of greater functional integrity with increased outer polyurethane coatings is clear.

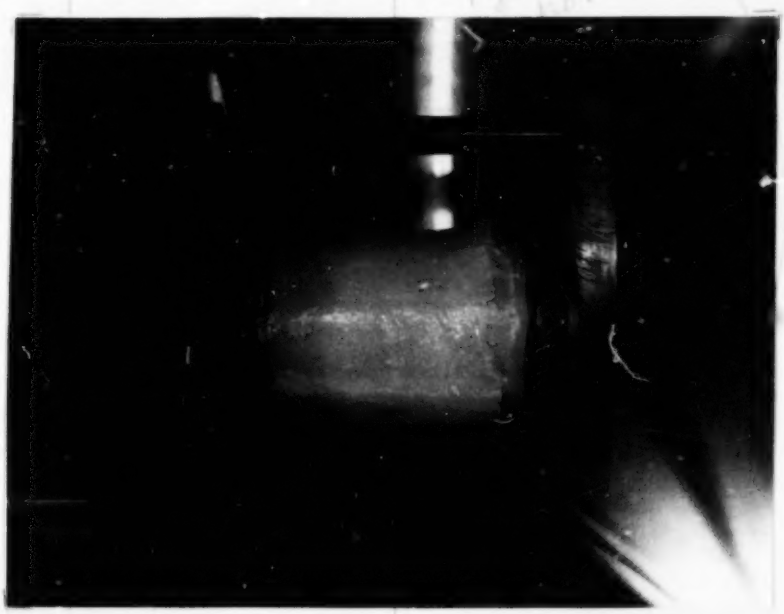

Figure 22. Encapsulate under compressive stress.

Figure 23. Coating integrity maintained during reinforcement failure (based on visual observation).

Figure 24. Failure mode of encapsulate (within lst 7% of deflection). Specimen does not appear to have been deflected to 7%; however, recovery to 80% to 90% of original dimension occurred after release of stress (except for those compressed to failure, beyond recovery).

Figure 25. Continuous deformation and drawing of thicker coating upon stress.

Figure 26 shows the load deflection curves for three hollow encapsulates with different coating thicknesses. (Hollow specimens were used to accentuate jacket properties.) The initial stiff modulus of each curve reflects the reinforcement's load bearing capabilities. At about 3% deflection, the reinforcements fail and a loss of load bearing capacity is evident. Failure of the coating is essentially coincidential with reinforcement failure for Specimen I. The added thickness of Specimen II shows stress transfer to the coating, and Specimen III provides more load-bearing capability. Clearly, as the coating thicknesses increase, the load-bearing properties reflect a trend toward tougher plastic behavior.

Figure 27 shows a load deflection curve for an encapsulate holding sand. The reinforcement's failure is noted at 3.0%, and the transfer of the load is presumably taken by the sand and jacket acting together until failure at ~19% compression.

Attempts to Produce Encapsulates with Thicker Coatings

The production of thicker coatings was an important processing limitation, as previously described. Thicker coatings were hindered by three primary obstacles: (1) thicker coatings required longer times for full cure, (2) the low viscosity of formulated coating limited the thickness per application, and (3) thicker coatings appeared to cure first at surfaces exposed to the atmosphere.

TABLE 11. VERTICAL COMPRESSION DATA FOR ENCAPSULATES

Specimen No. \ Property	No. of Applied Coats	Estimated Coating Thickness, mm (in.)	Reinforcement Yield kN, (lb)	Deflection @ Reinforcement Yield, %	Deflection @ Jacket Failure, %
1	1	0.33 (0.013)	12.54 (2820)	2.9	2.9
2	2	0.66 (0.026)	12.46 (2800)	3.0	3.0
3	4	1.32 (0.052)	11.97 (2690)	3.1	4.0
4	5	1.65 (0.065)	12.68 (2950)	3.2	7.1
5	7	2.31 (0.091)	12.28 (2760)	3.0	12.2
6	9	2.97 (0.117)	14.63 (3290)	3.3	19.8

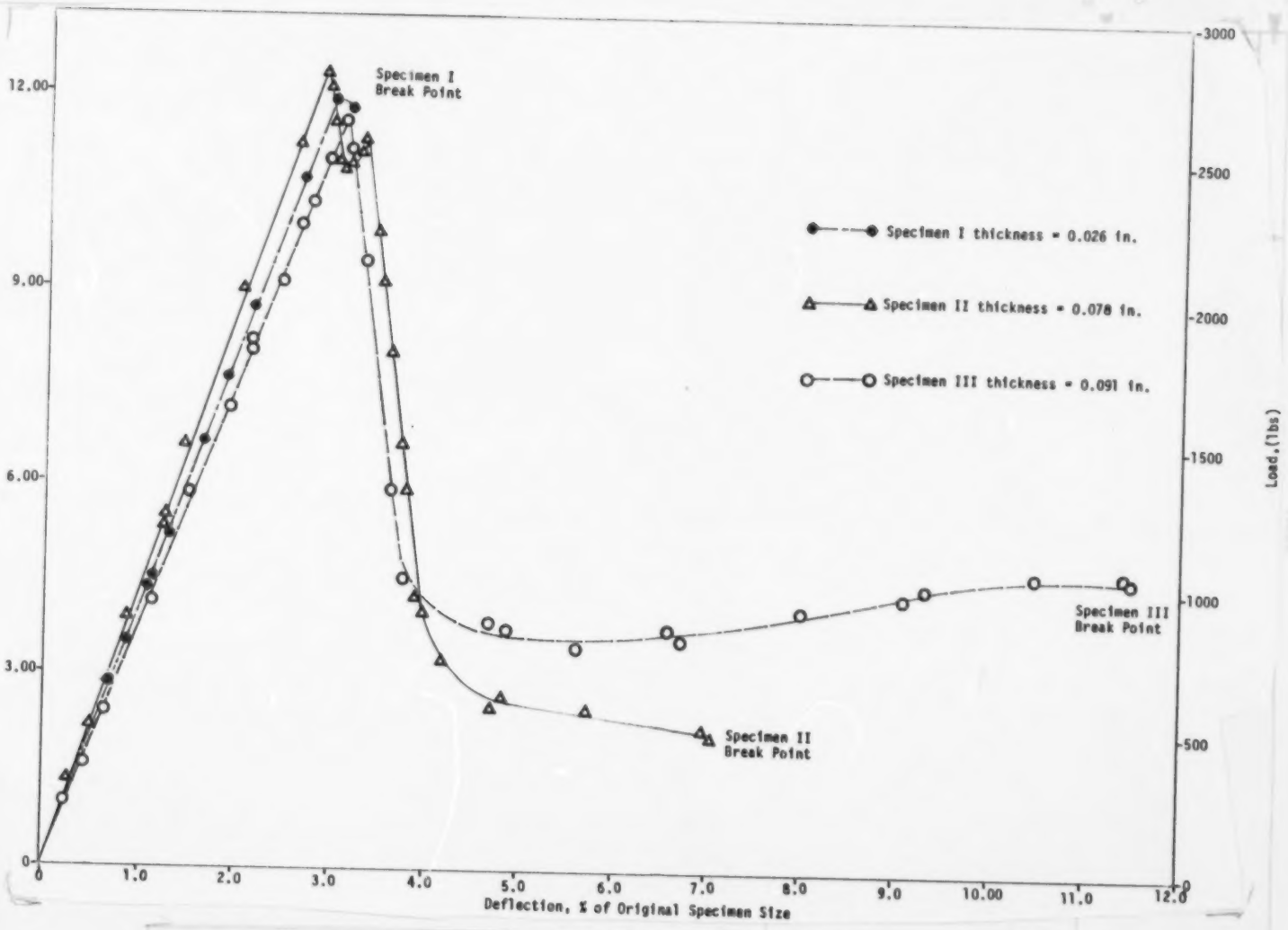

Figure 26. Load-deflection responses of three hollow encapsulates with successively thicker coatings.

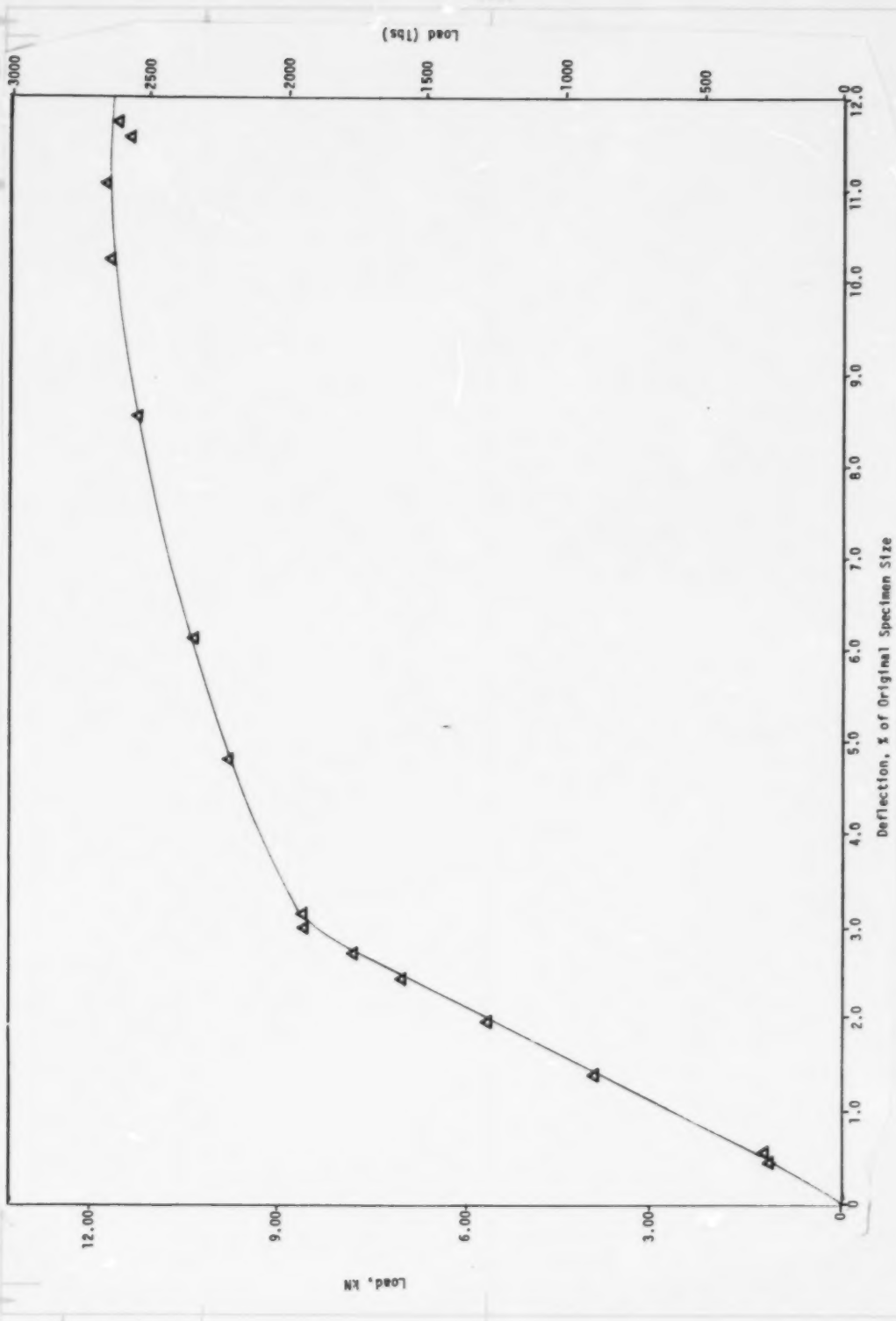

Figure 27. Load-deflection response of sand-filled encapsulate.

Attempts to mitigate these problems involved evaporation of water before coating application. Table 12 lists results of an experiment in which increases in viscosity and reduction of water content were accomplished. Samples of Neorez R-960 and the formulated resin were placed in wide-mouth jars and allowed to free-stand with periodic mixing. (Samples left free-standing without agitation began to gel.)

The loss in water content after 4 to 8 hr was probably not significant in view of the remaining portion left to evaporate for full cure. However, the increases in viscosity were helpful for achieving thicker coatings. Coatings formed with initial viscosities of ~0.440 Pa·s (~440 cps) had resin run-off (down the encapsulate's walls) similar to that observed when attempting to apply thicker coatings of paint. Viscosities >2.000 Pa·s (>2000 cps) aided by eliminating some run-off.

The technique found to be most convenient and leading to thickest coatings involved evaporation of water from the R-960 to viscosities >4.000 Pa·s (>4000 cps), followed by formulation and application. But this technique was sensitive to foam formation and its entrapment in the viscous resin. Removal was accomplished as described in Section 5, although it was more difficult.

Curing first at surfaces exposed to the atmosphere was initially observed during the hardness studies described in Section 6. Though this was not a serious processing problem in the range of thicknesses achieved on lab specimens,* it should be recognized as a potential limitation affecting cure time.

Overall, single applications of coatings were achieved that were up to 0.38 mm (~0.015 in.) thick, but not without the penalty of curing times, which were 2 to 3 times longer.

LEACHING TESTS

The purpose of leaching tests was to evaluate performance capabilities of encapsulates exposed to ambient aqueous solution stresses. Encapsulates were submerged in deionized water baths, with leachate analyses conducted periodically. This work, described in detail below, indicated high localization potential.

Specimen Preparation and Testing

Six fiberglass casings were charged to capacity with a waste simulant, sodium chloride, and coated with two layers of polyurethane (ensuring complete sealing). All curing was achieved at laboratory temperatures (22° to 23°C), allowing 4 hr of curing between each coating application. Encapsulates were given a thorough wash in deionized water.

*Specimens for hardness studies were ~6.35 mm (~0.25 in.) thick, formed by casting formulated resins into aluminum dishes. The preparation of coatings on encapsulates with this range of thickness was not achieved.

TABLE 12. VISCOSITY* - WATER LOSS STUDY

Property / Trade Name	Water Content (%)				Viscosity, Pa·s (cps)				
	Initially	4 hrs	8 hrs	20 hrs	Initially	4 hrs	8 hrs	20 hrs	72 hrs
Neorez R-960	66.1	65.0	64.2	-	0.445 (445)	2.000 (2000)	4.200 (4200)	8.400 (8400)	70.000 (70,000)
Formulated Coating	66.0	65.5	64.7	-	0.440 (440)	1.100 (1100)	Not manageable due to gelation		

*Brookfield Viscometer, Model LTV

Polyethylene leaching baths were prepared by washing to eliminate any contamination. Washing included tap water rinse with brushing, Alconox wash with brushing, deionized water rinse, 1:1 nitric acid, and three thorough deionized water rinses.

Each encapsulate was immersed in 1500 ml of deionized water leaching solution and was routinely agitated during the testing period (Figure 28). Leachate sampling and analyses occurred after 7, 30, and 60 days.

Each bath was sampled by withdrawing 25.00 ml of leachate. Concentration effects involved by sample withdrawal were considered when calculating chloride concentrations. Since the volume of leachate removed during the entire period was relatively small (225 ml), no additional water was added to the baths.

Leachate analyses for chloride consisted of quantitative titration of 25.00-ml samples with primary standard silver nitrate (0.0140N) to a visual end point. Three titrations per bath were conducted to assure proper end-point detection. Deionized water was used as a blank for all determinations.

Experimental Results

Table 13 lists the analytical data. Those values reported as <1.5 ppm correspond to the estimated lower detection limit value for the technique.[12] The chloride level for No. 5 was 12.4 ppm; since it was essentially con-

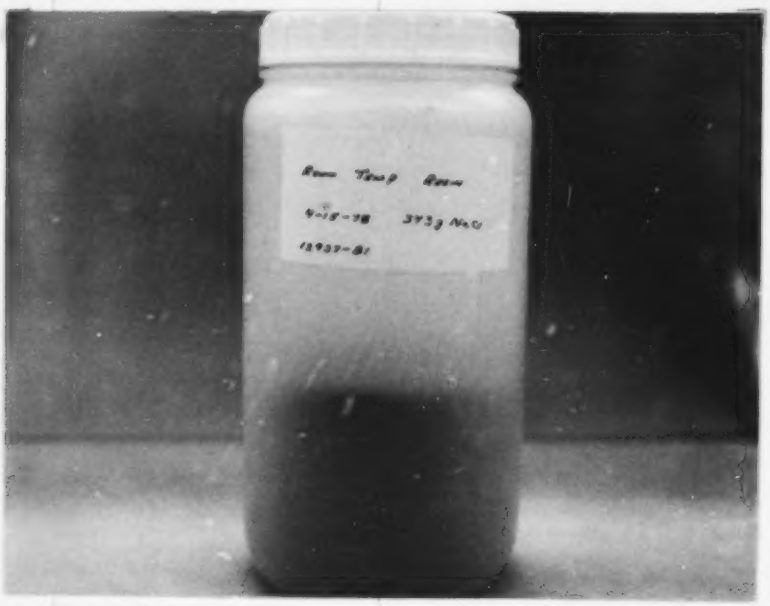

Figure 28. Test specimen in aqueous leaching bath.

TABLE 13. CONCENTRATION OF CHLORIDE (ppm) FOR SPECIMENS IMMERSED IN DEIONIZED WATER LEACHING SOLUTION

Sample No. \ Immersion Period (Days)	7	30	60	Maximum Loss* of Chloride
1	<1.5**	<1.5	<1.5	143,000
2	<1.5	<1.5	<1.5	143,000
3	<1.5	<1.5	<1.5	143,000
4	<1.5	<1.5	<1.5	143,000
5	12.41	12.43	12.43	143,000
6	<1.5	<1.5	<1.5	143,000

*These are approximate concentrations (derived by calculation) assuming complete failure of the specimen and subsequent contamination of the leaching solution.

**This is the estimated lower detection limit value of the technique employed.[12]

stant throughout the study it was attributed to surface contamination at the onset of immersion.

Those values reported under "maximum loss of chloride" were expected concentrations derived by calculation and assuming complete failure of encapsulates and subsequent contamination of leaching solution. Within detection limits, these comparative data clearly demonstrate the effectiveness of encapsulation against chloride loss.

All analytical methods and procedures can be found in References 12 and 13.

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SECTION 9

RESULTS AND DISCUSSION

This report demonstrates a means for encapsulating containerized hazardous wastes (primarily 55-gal drums) at atmospheric temperatures. In particular, a bench-scale fabrication technique was developed (Section 7) whereby test specimens holding consignments of sodium chloride (simulating heavy-metal-containing, water soluble salts) were produced and subjected to leaching and compressive stresses. A high localization capability by encapsulation is clear from the leaching data reported in Table 13. The specimens were found to resist failure when compressed to about 80% of their original heights (Section 8). Under greater compression, the encapsulate jackets underwent rupture (Figure 22), and reasons for rupture were shown to be related to the thickness of the outer polyurethane jackets (Figure 26).

Accomplishment of encapsulation at atmospheric temperatures involved jacketing specimens with a novel water-based polyurethane coating (Figure 15) in advance of state-of-the-art counterparts. Consequently, some characterization and evaluation studies (Sections 4, 5, and 6) were conducted and resulted in a formulation and pigmentation system suitable for spray-on or brush-on operations.

The results of this work led to the realization of several attractive features and disadvantages of the process.

The principal features of the process are:

- No need exists for applied thermal energy, as with thermoplastics and thermosets.
- State-of-the-art spray-on equipment for resin application may be employed directly.
- A method is offered for managing distorted 208-liter (55-gal) drums (however, this advantage may adversely affect subsequent handling techniques).
- No need exists for heavy-duty or high-tolerance equipment.
- The technique is viewed to be generally applicable without tailoring the process to contents of drums (wet or dry wastes).

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The principal disadvantages of the process are:

- Use of a special, higher priced, marginally characterized resin is required.
- Special safety equipment is required by personnel during formulation/fabrication because of toxicity of cross-linker (Appendix B).
- Formation of thick coatings requires multiple applications.
- Coatings require longer curing times at lower atmospheric temperatures.

Of the disadvantages listed, the overriding limitation affecting feasibility is the inability to achieve thicker coatings with one or a few applications. A minimal number of applications is desirable because of reduced man-hour requirements.

Mitigation of the above deficiency would require modification of the advanced resin formulation (Section 5). This resin modification would inherently define several new areas of concern:

- Identification of one or more additives (possibly thixotropic) that would enhance thicker coatings and not hinder curing.
- Identification of compatible pigmentation system (thixotropic agent and pigment might be single component).
- Demonstration that altered formulation is manageable with state-of-the-art spray-on equipment.
- Demonstration that altered formulation does not degrade coating properties or encapsulate performance.

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APPENDIX A

MECHANICAL TESTING

A. Tensile

1. All tests were conducted on an Instron Universal Testing Instrument, model 1130.

2. Testing was conducted in accordance with ASTM-D-412.

3. Testing speed = 2 in. per min.

4. Statistical Data on tables was calculated according to Skoog, D. S., West, D. M., Fundamentals of Analytical Chemistry, 2nd Ed., p. 38, Holt, Rinehart and Wilson, Inc., New York (1971).

B. Hardness

1. Shore A

   a. Samples were cured for 24 hr at room temperature (23° to 24°C).

   b. Testing was conducted in accordance to ASTM-D-2240.

   c. P.T.C. Durometer, model 306, Type A.

2. Pencil

   a. Samples were cured for 24 hr at room temperature (23° to 24°C).

   b. Testing was conducted in accordance with Military Specification C-83286B (USAF), Amendment 1, (1975).

C. Immersion Tests

1. Resistance of plastic films to extraction by chemicals.

   a. Samples cured for 24 hr at 23° to 24° C before immersion.

   b. Testing was conducted in accordance with ASTM-D-1239, part 4.1.

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D. Compression Tests

1. All tests were based on ASTM-C-39-71, but modified to accommodate specimen size.

2. Initial testing was conducted on a Cal Tester, Model TH-5, and quantitative data were obtained using a Reihle Testing Machine, Model F-5-160.

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APPENDIX B

TOXICITY DATA FOR CX-100

An acute oral toxicity study with CX-100 produced an LD_{50} value of 3,038 mg/kg. This places it in a category of toxic materials with a value which is similar to common solvents such as methyl ethyl ketone and toluene.

A primary skin irritation value of 2.65 was obtained out of a possible score of 9. Compounds producing a primary irritation value of 5 or greater are defined as "primary skin irritants".

CX-100 was found to be a primary eye irritant, and corrosive effects were indicated.

CX-100 was tested for potential mutagenic activity in microbial assays using the reverse mutation technique described by Ames. A dose-related increased reversion frequency over the spontaneous rate was obtained and a value of 0.18 revertants per microgram was calculated. Although positive, the level of response is measurably low.

Mixtures of NeoCryl A-601 latex with the recommended amount of CX-100 were tested immediately upon mixing, and at various elapsed times. None of the mixtures showed any genetic response in the Ames Test. This indicates that the coating vehicle containing CX-100 before and during application does not have any measurable mutagenic activity, although the title compound, CX-100, is mildly active.

Films of NeoRez R-960 and NeoCryl A-601 containing recommended amounts of CX-100 crosslinker did not demonstrate any mutagenic activity in any of the assays tested. It can, therefore, be concluded that dried films incorporating CX-100 as a crosslinker can be considered non-mutagenic by the test conditions described by Ames.

HANDLING

Care must be taken to avoid any contact with the skin and eyes. When used in spray applications, particular care must be taken to avoid oral and/or nasal ingestion.

Where spillage of CX-100 occurs, the area should be thoroughly flushed with water. This will hydrolyze the CX-100 and make it easier to clean. If more rapid cleaning is desirable, a 5% solution acetic acid or a mineral acid can be used.

Containers of CX-100 must show the following warning:

HANDLE WITH CARE

USE ONLY WITH ADEQUATE VENTILATION. AVOID CONTACT WITH SKIN OR EYES AND BREATHING OF SPRAY MIST. IN CASE OF SKIN OR EYE CONTACT, FLUSH THOROUGHLY WITH PLENTY OF WATER. FOR EYES, ALSO CALL PHYSICIAN. CLOSE CONTAINER AFTER EACH USE. KEEP OUT OF REACH OF CHILDREN. STORE IN COOL, DARK PLACE.

APPENDIX C

ENCAPSULATION OF UNCONFINED WASTES AT JBF SCIENTIFIC (BOSTON) LABORATORY

JBF Scientific supplied waste samples of the following compositions for demonstration of applicability of the encapsulation process:

1. Dry Solid

NaCL	44%
Na_2SO_4	40%
Organic Arsenicals	2%

2. Wet Solid

Filter Acid (Celite)	33%
Phosphoric Acid	42%
Arsenic Trisulfide	2%
Maybe Activated Carbon in Waste	

3. Dry Powder

90% Pure Arsenic Trioxide

The wastes were managed in a three-step procedure. The products of each step are given in Figure C-1.

The waste indicated by #1 was readily encapsulated at JBF by the Process. This waste was previously managed under EPA contract and was reported in EPA-600/2-77-045 August 1977. This report also gives details of the encapsulation process. Waste #2 was also similarly managed and successfully encapsulated.

Waste #3 was managed differently. Because of the high vapor pressure of this waste at elevated temperatures, this waste was agglomerated by Portland cement instead of resin. The agglomerate was then encapsulated by polyethylene. This work represented the first demonstration of polyethylene encapsulation of cement-hardened wastes.

The encapsulated wastes remained at JBF for evaluation in their program for EPA, Cincinnati.

Figure C-1. Product of each step for encapsulation left to right: A) agglomerated waste particulates, B) polyethylene jacketed agglomerate bottom and sides, and C) polyethylene encapsulate.

APPENDIX D

COMPARATIVE ANALYSIS OF HAZARDOUS WASTE MANAGEMENT OPTIONS

The chart in this section provides a comparative analysis of waste management options stemming from work for EPA by TRW and those being investigated or in practice. It should be recognized that wastes managed here are not manageable by some of the other means. But at least the costs can be compared for the purpose of showing that the new options do not incur costs that differ significantly from the norm.

TABLE D-1

COMPARATIVE ANALYSIS OF HAZARDOUS WASTE MANAGEMENT OPTIONS

TRW AND OTHER MANAGEMENT SCHEMES ★ (MAJOR TREATABLE WASTES)	ESTIMATED COSTS[2] FOR SECURING WASTES	COST/FT[3] OF WASTE	COMMON TREATABLE WASTE TYPES	CURRENT STAGE OF DEVELOPMENT	ADVANTAGES	DISADVANTAGES	COMMENTS
ENCAPSULATION							
RESIN FUSION PROCESS[b] (UNCONFINED PARTICULATES AND SLUDGES) (AT TRW UNDER EPA SPONSORSHIP)	$110/TON OF DRY WASTE (BASED ON 20,000 TONS/YEAR)	$5.06[l]	UNCONFINED PARTICULATES AND SLUDGES OF TOXIC AND SOLUBLE INORGANICS AND OTHER COMPOUNDS, ALL WASTES LISTED UNDER FIXATION, WASTES NOT MANAGEABLE BY OTHER MEANS	LABORATORY R&D COMPLETED	VERY HIGH PERFORMANCE PRODUCTS, SIMPLE EQUIPMENT, GENERAL APPLICABILITY TO MANY WASTE TYPES	HEAT REQUIRED TO FUSE RESIN, REQUIRES PARTICULATED WASTES	BROADER APPLICABILITY TO WASTE TYPES THAN THAT REALIZED BY COMPETING SYSTEMS, EXPECTED TO COMPLY WITH DOT REGULATIONS GOVERNING TRANSPORTING OF HAZARDOUS WASTES
RESIN FUSION PROCESS[c] (55-GAL DRUMS AND SMALLER CONTAINERS) (AT TRW UNDER EPA SPONSORSHIP)	$0.45/GAL	$3.375[m]	PRIMARILY 55-GAL DRUMS AND SMALLER CONTAINERS OF WASTES,	LABORATORY R&D COMPLETED	CONTAINERIZED WASTES SECURED IN SEAM-FREE, ENCOMPASSING PLASTIC JACKET, VERY HIGH MECHANICAL AND CHEMICAL PERFORMANCE, SIMPLE EQUIPMENT	HEAT REQUIRED TO FUSE RESIN, EQUIPMENT NOT COMMERCIALLY AVAILABLE	EXPECTED TO YIELD ENCAPSULATES WITH GREATER PERFORMANCE THAN THAT OBTAINABLE BY STATE-OF-THE-ART TECHNIQUES, EXPECTED TO COMPLY WITH DOT REGULATIONS GOVERNING TRANSPORTING OF HAZARDOUS WASTES
CEMENTITIOUS ENCAPSULATION WITH MATERIALS AVAILABLE TO THE PUBLIC[c] (SMALL CONTAINERS) (AT TRW UNDER EPA SPONSORSHIP)	$0.60/ENCAPSULATE (BASED ON RAW MATERIALS ONLY)	$0.90	SMALL CONTAINERS OF WASTE	PREPARING INSTRUCTIONS FOR PUBLIC USE	EMPLOYS LOW COST MATERIALS AND EQUIPMENT READILY AVAILABLE TO THE PUBLIC, MATERIAL USE IS FAMILIAR, WELL KNOWN TECHNIQUE	SUITABLE FOR SMALL CONTAINERS ONLY, SUSCEPTIBLE TO DEGRADATION UNDER ACIDIC LANDFILL CONDITIONS, REQUIRES FORMULARY ADJUSTMENT BY USER	DESIGNATED FOR USE BY SMALL MANUFACTURERS, SCHOOLS, HOSPITALS, PHARMACEUTICAL AND BIOLOGICAL LABS, SMALL GENERATORS OF PESTICIDES, HERBICIDES, FUNGUSCIDES
RESIN SPRAY-UP PROCESS[c] (55-GAL DRUMS) (AT TRW UNDER EPA SPONSORSHIP)	COST NOT YET DETERMINED	—	ALL WASTES LISTED UNDER RESIN FUSION PROCESS FOR CONTAINERS OF WASTES (EXCLUDING SMALLER CONTAINERS)	LABORATORY R&D INCOMPLETE	UTILIZES COMMERCIALLY AVAILABLE SPRAY-UP EQUIPMENT AND TECHNIQUES WITH WHICH THERE IS GENERAL FAMILIARITY, NO HEATING REQUIRED TO THERMOSET RESIN	REQUIRES CUSTOM FORMULATED RESINS, NOT SUITABLE FOR SMALL CONTAINERS	SPRAY-UP PROCESS MAY REQUIRE USE OF HIGHER COST RESINS THAN THOSE EMPLOYED IN THE RESIN FUSION PROCESSES
CEMENTITIOUS ENCAPSULATION WITH READY-MIX CONCRETE[c] (SMALL CONTAINERS) (AT TRW UNDER EPA SPONSORSHIP)	$3.30/ENCAPSULATE (BASED ON RAW MATERIALS ONLY)	$4.95	SMALL CONTAINERS OF HAZARDOUS AND LOW LEVEL RADIOACTIVE WASTES	PREPARING INSTRUCTIONS FOR PUBLIC USE	EXPECTED TO YIELD HIGH PERFORMANCE PRODUCTS WHICH ARE STABLE UNDER ACIDIC LANDFILL CONDITIONS, DOES NOT REQUIRE FORMULARY ADJUSTMENT BY USER	SUITABLE FOR SMALL CONTAINERS ONLY	CAN BE AVAILABLE PRE-PACKAGED AND READY FOR USE WITH ADDITION OF ONLY WATER
PLASTIC WELDING PROCESS[c] (55-GAL DRUMS AND SMALLER CONTAINERS) (AT TRW UNDER EPA SPONSORSHIP)	$253/TON (BASED ON 80,000 55-GAL DRUMS/YEAR)	$8.13[n]	PRIMARILY 55-GAL DRUMS AND SMALLER CONTAINERS OF ALL WASTES LISTED UNDER RESIN FUSION PROCESSES, FIXATION, OVER-PACKS	FABRICATION OF FULL SIZE PROTOTYPES	SEAM-FREE PLASTIC ENCAPSULATE, STABLE UNDER CORROSIVE ENVIRONMENTAL CONDITIONS, LOW COST AND SIMPLE EQUIPMENT, EXPECTED TO BE TRANSPORTABLE IN COMPLIANCE WITH DOT REGULATIONS GOVERNING TRANSPORTING OF HAZARDOUS WASTES	REQUIRES AN APPARATUS NOT COMMERCIALLY AVAILABLE	EXPECTED TO YIELD PLASTIC OVER-PACKS FOR 55-GAL DRUMS WITH PERFORMANCE SIGNIFICANTLY IN ADVANCE OF CURRENTLY EMPLOYED 55-GAL STEEL OVER-PACKS
OVER-PACKS[d] (55-GAL DRUMS)	$243/TON (BASED ON 80,000 55-GAL DRUMS/YEAR)	$7.82[n]	55-GAL DRUMS OF HAZARDOUS WASTE	COMMERCIAL	SIMPLE DESIGN, DEVELOPED TECHNOLOGY, MINIMAL CAPITAL EQUIPMENT INVESTMENT, MINIMAL LABOR REQUIREMENTS	STEEL OVER-PACKS ARE SUBJECT TO APPRECIABLE CHEMICAL CORROSION, PERFORMANCE CAN BE SIGNIFICANTLY AFFECTED BY SMALL DIMENSIONAL DISTORTIONS	CURRENTLY USED FOR SECURING LEAKING BARRELS OF HERBICIDE WASTES AT MANUFACTURER'S SITES, USE FOR TRANSPORTING OF HAZARDOUS WASTE UNDER QUESTION

INCINERATION							
LAND-BASED[e]	$179-$210/TON	$5.78-$6.78[n]	HALOGENATED ORGANIC LIQUIDS INVESTIGATIONS UNDERWAY CONCERNING ORGANIC SOLIDS	COMMERCIAL	CAN COMPLETELY DESTROY WASTES, WELL KNOWN TECHNOLOGY, HIGH VOLUME CAPACITY	PRODUCES NON-COMBUSTIBLE TOXIC ASH, REQUIRES SCRUBBING OF EFFLUENT, EQUIPMENT SUBJECT TO APPRECIABLE CORROSION	UNFAVORABLE PUBLIC REACTION IN PLACING INCINERATION SITES
AT-SEA[e]	$79.3-$90.2/TON	$2.47-$2.81[n]	SIMILAR TO LAND-BASED INCINERATION	COMMERCIAL	SAME AS LAND-BASED INCINERATION BUT REQUIRING LESS STRINGENT SCRUBBING OPERATIONS	PRODUCES NON-COMBUSTIBLE TOXIC ASH, EQUIPMENT SUBJECT TO APPRECIABLE CORROSION	ALLEVIATES SOME PUBLIC HOSTILITY ENCOUNTERED WITH LAND-BASED SITES
FIXATION							
CEMENT-BASED[f]	$0.03-$0.04/GAL (1978)[f]	$0.225-$0.30[m]	NON DESTRUCTIBLE WASTES AND WASTES ECONOMICALLY INFEASIBLE TO TREAT BY OTHER MEANS	MANY PROCESSES ARE COMMERCIAL	LOW COST, USES CEMENT EQUIPMENT	WEIGHT ADDED IN FIXING WASTES IS APPRECIABLE, LOW PERFORMANCE PRODUCTS, PRETREATMENT REQUIRED TO MAKE WASTES SUITABLE FOR FIXATION	CURRENT TRENDS ARE TO SECURE WASTES WITH MINIMAL AMOUNTS OF FIXATION ADDITIVE IN ORDER TO CONSERVE LANDFILL SPACE
LIME-BASED[f]	$0.03-$0.05/GAL (1978)[f]	$0.225-$0.375[m]					
ORGANIC-POLYMER[f]	$647/TON[m]	$20.90 (1978)[f]			YIELDS HIGH PERFORMANCE PRODUCTS, DOES NOT REQUIRE DEWATERING OF WASTES	APPRECIABLE COST, RESIN THERMOSETTING REACTION SENSITIVE TO IMPURITIES	IN PILOT PLANT STAGE OF DEVELOPMENT
THERMOPLASTIC[f]	COST AVAILABLE ON CASE-BY-CASE BASIS ONLY	——			DOES NOT REQUIRE CHEMICAL REACTION TO HARDEN RESIN	HEAT REQUIRED TO MELT RESIN, DIFFICULT TO MIX IN HIGH QUANTITIES OF WASTE MATERIALS	PERFORMANCE DEPENDS UPON TERMOPLASTIC RESIN SELECTED
DEEP WELL INJECTION[g]	$150,000/YEAR TO MAINTAIN AND $0.02/GAL FOR ENERGY (ASSUMING 1 MILE DEEP, 6 MILLION GAL/MONTH)	$0.0158[p]	PRIMARILY LIQUIDS	COMMERCIAL	LOW COST, HIGH VOLUME CAPACITY	REQUIRES SUITABLE GEOLOGICAL FORMATIONS, OPERATIONS SENSITIVE TO SOLIDS IN WASTE WHICH CAN PLUG UP FLOW	STABILITY OF WASTES IN DEEP WELL BEING QUESTIONED
BIOLOGICAL DEGRADATION[h] (COMPOSTING OR LAND-FARMING)	$16.50/TON (BASED ON OILY WASTES)	$0.5148[p]	SOME ORGANICS AND BIOCHEMICALS	COMMERCIAL	READILY EMPLOYED, MAY LEAVE UTILIZABLE RESIDUE	NOT APPLICABLE TO DETOXIFY HEAVY METAL WASTES	REQUIRES STRINGENT CONTROLS TO REALIZE EFFECTIVE WASTE CONVERSION
PYROLYSIS[i, j]	——	——	——	——	DOES NOT SIGNIFICANTLY CONTRIBUTE TO CO_2 POLLUTION IN ATMOSPHERE	REQUIRES APPRECIABLE THERMAL ENERGY, MAY RELEASE NOXIOUS GASES	——
WASTE OXIDATION WITH MOLTEN SALTS[i, k]	——	——	PRIMARILY HALOGENATED WASTES	LABORATORY R&D	EXCELLENT HEAT EXCHANGE CHARACTERISTICS, MAY NEUTRALIZE HALOGENS IN MOLTEN SALT REACTIONS	LEAVES CONTAMINATED SPENT SALTS THAT MUST BE DISPOSED OF OR RECYCLED, MAY PRODUCE NOXIOUS GASES	R&D STAGES OF DEVELOPMENT
ELECTRO-MAGNETIC (PLASMA) WAVE MODIFICATION	——	——	PRIMARILY HALOGENATED WASTES	LABORATORY R&D	——	——	VERY LITTLE PERTINENT LITERATURE IDENTIFIED
VAULT ENCAPSULATION	——	——	——	——	HIGH WASTE CAPACITY	INVOLVES MOVING UNSECURED WASTES TO VAULTS	EXPERIMENTAL SYSTEM

*Current Vendor: Environmental Protection Polymers, Inc., 13414 Prairie Avenue, Hawthorne, CA 90250.

a. 1979 DOLLARS, EXCEPT WHERE NOTED OTHERWISE. b. "DEVELOPMENT OF A POLYMERIC CEMENTING AND ENCAPSULATING PROCESS FOR MANAGING HAZARDOUS WASTES," EPA-600/2-77-045. c. "LABORATORY AND FIELD EVALUATION OF PROCESSES AND/OR MATERIALS TO ENCAPSULATE WASTE/CONTAINERS OF NON-RADIOACTIVE HAZARDOUS MATERIALS," EPA CONTRACT NO. 68-03-2483. d. PERSONAL COMMUNICATIONS WITH REPRESENTATIVES FROM (1) ROUND CONTAINER COMPANY, INC., CHARGRIN FALLS, OH, (2) CLEARING CONTAINER, INC., CHICAGO, IL, (3) VERTAC, JACKSONVILLE, AK, (4) MIREX CORP., INGLEWOOD, CA, 6/79. e. "COMPARATIVE COST ANALYSIS AND ENVIRONMENTAL ASSESSMENT FOR DISPOSAL OF ORGANOCHLORINE WASTES," EPA-600/2-78-190. f. POJASEK, R., ED., "TOXIC AND HAZARDOUS WASTE DISPOSAL," VOL. 1. ANN ARBOR SCIENCE PUBLISHERS INC., ANN ARBOR MICH., 1979. g. SMITH, M.E., "SOLID-WASTE DISPOSAL: DEEPWELL INJECTION," CHEMICAL ENGINEERING, McGRAW-HILL, N.Y., 4-9-79. h. HUDDLESTON, R.L., "SOLID-WASTE DISPOSAL: LAND FARMING," CHEMICAL ENGINEERING, McGRAW-HILL, N.Y., 2-26-79. i. MARYNOWSKI, C.W., "DISPOSAL OF POLYMER SOLID WASTES BY PRIMARY POLYMER PRODUCERS AND PLASTICS FABRICATORS," EPA CONTRACT NO. PH-68-68-160, 1972. j. HIRASKA, M., "BEHAVIOR OF HAZARDOUS SUBSTANCES IN STABILIZATION AND SOLIDIFICATION PROCESSES OF INDUSTRIAL WASTES," ACS, 177TH NATIONAL SYMPOSIA, APRIL, 1979. k. POJASCK, R., "DISPOSAL OF HAZARDOUS CHEMICAL WASTES," ENVIRONMENTAL SCIENCE AND TECHNOLOGY, 7/79. l. ASSUMING WASTE DENSITY OF 92 LB/FT^3. m. ASSUMING WASTE DENSITY OF 7.5 GAL/FT^3. n. ASSUMING WASTE DENSITY OF 64.6 LB/FT^3. p. ASSUMING AN OILY WASTE DENSITY OF 62.4 LB/FT^3.